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OUTLINES OF ENVIRONMENTAL PARAMETERS

COORDINATED BY WATER QUALITY SECTION

LABORATORY SERVICES BRANCH

APRIL 1987

Ministry
of the
Environment

G.C.RONAN,Director

Laboratory Services Branch

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OUTLINES OF ENVIRONMENTAL PARAMETERS

A guide to the occurrence, significance, sampling and analysis of chemical and microbiological parameters in water, sediment, soil, vegetation and air.

COORDINATED BY

Vera Turner and Peter Campbell

WATER QUALITY SECTION

Ontario Ministry of the Environment

Laboratory Services Branch

June, 1987.

FOREWORD

Information on environmental parameters and analytical methods is often required in order to choose the most suitable tests for environmental problems, to correctly interpret data and to prepare reports. This summary has been prepared in order to provide information in a brief, non-technical form and to assist the sampler in understanding chemical and microbiological parameters as well as their limitations. Performance reports, outlining quality control protocols and providing data for individual parameters may be obtained from individual Sections at Laboratory Services Branch (LSB): Inorganic Trace Contaminants Section (ITC), Water Quality Section (WQ), Trace Organics Section (TO) and Drinking Water Organics Section (DWO).

Methods in this manual are suitable for routine chemical and microbiological analyses on several sample matrices and include specific information as to proper sampling and preservation techniques. Non-routine tests, not listed in this manual, may be performed by laboratory staff upon special request. Information about these tests may be obtained from the Laboratory Services Branch, Ontario Ministry of the Environment, Box 213, Rexdale, Ontario, M9W 5L1, (see Table 1 for contact names).

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Trace Organics Section: George Crawford and Wendy Moss.

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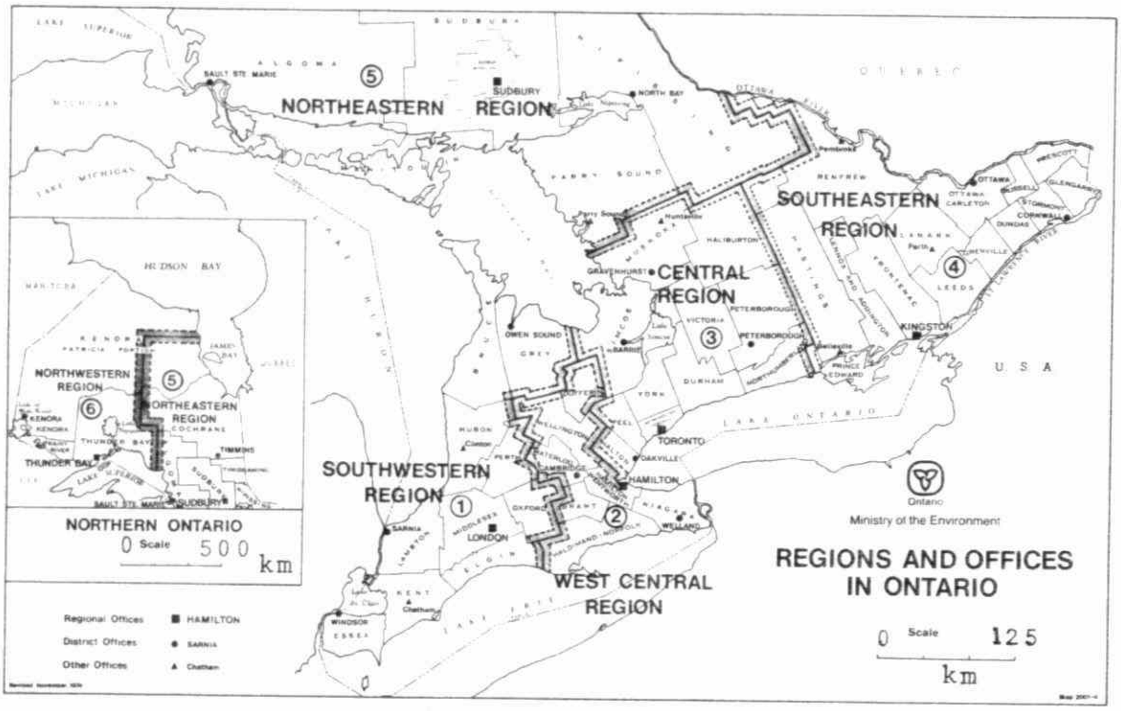
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NORTH BAY
SOUTHEASTERN REGION
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CORNWALL
KINGSTON
Pembroke
Perth
CENTRAL REGION
③
PETERBOROUGH
GRAVENHURST
Huntsville
BARRIE
TORONTO
OAKVILLE
SOUTHWESTERN REGION
①
LONDON
SARNIA
WINDSOR
Chatham
Clinton
OWEN SOUND
WEST CENTRAL REGION
②
HAMILTON
CAMBRIDGE
WELLAND
LAKE ONTARIO
LAKE ERIE
LAKE HURON
LAKE SUPERIOR
LAKE MICHIGAN
QUEBEC
U.S.A.
NORTHERN ONTARIO
NORTHWESTERN REGION
⑥
KENORA
THUNDER BAY
TIMMINS
HUDSON BAY
0 Scale 500 km
Regional Offices ■ HAMILTON
District Offices ● SARNIA
Other Offices ▲ Chatham
Ontario
Ministry of the Environment
REGIONS AND OFFICES IN ONTARIO
0 Scale 125 km

INTRODUCTION

The various laboratories of the Laboratory Services Branch are equipped to perform a large number of standard chemical and microbiological analyses on domestic water supplies, surface waters, ground waters, domestic and industrial wastes as well as air, soil, sediment, vegetation and sludge samples. Special analyses required for research studies and unusual pollution problems may also be provided upon consultation with laboratory staff.

Three regional laboratories at Kingston, London and Thunder Bay are operational in addition to the main Toronto Laboratory. Analytical methods used at these laboratories may differ slightly in detail from the Main Laboratory but results are comparable. The boundaries of the districts serviced by these laboratories are shown in Figure 1. Samples collected in Regions 2, 3 and 5 are analyzed in Toronto. Samples collected in Regions 1, 4 or 6 are analyzed in London, Kingston and Thunder Bay respectively when the analytical capability is available. If such capability is not available, these samples are analyzed in Toronto.

INSERT
FIG. 1
HERE

Figure 1

The tests performed by each regional laboratory as well as the sectional laboratories in Toronto are outlined in "A Guide to the Collection and Submission of Samples for Laboratory Analysis (1985); **(hereafter called the "Sampling Guide")**, available from the Water Quality Section, Laboratory Services Branch, MOE, Box 213, Rexdale, Ontario M9W 5L1. Field samplers should ensure that a laboratory is capable of analyzing the parameter(s) requested before shipping. Mobile laboratories or field stations, such as at Dorset, may perform a limited number of tests in conjunction with major surveys or studies.

Collection and Perishability of Samples

The method of collecting the sample in the field is the concern of the individual in charge of the particular survey. If the sample is not representative of the whole, the subsequent analyses will be worthless. Care must be taken to prevent contamination of the sample by sampling devices, motor exhausts, disturbing the sediment beds of lakes etc. Furthermore, it must be realized that the composition of most samples will change with time; thus the sample must be quickly forwarded to the laboratory for analyses. The perishability of a sample may be due to biological activity, chemical precipitation, leaching of contamination from containers, adsorption on the walls of containers, chemical reactions between constituents and volatility. Tests such as dissolved oxygen, dissolved carbon dioxide, free chlorine and pH are examples of extremely perishable parameters. Field analyses for these parameters are recommended whenever possible.

For some parameters, the use of a preservative or refrigeration is recommended. These techniques may delay perishability somewhat, but the sample should still be transported to the laboratory as quickly as possible.

For samples which do not have a preservative already in the bottle, rinsing both the bottle and cap with sample two or three times is recommended for inorganic tests only. This procedure helps to equilibrate the sample with the container walls and hence "container effects" such as leaching or adsorption can be minimized. This procedure may also help reduce contamination. Samples to be tested for organic parameters should be collected without rinsing, since sample adsorption may occur at each rinse and the whole container may be extracted for analysis.

In view of the probable chemical degradation of a sample, the laboratory analyzes each sample as rapidly as present facilities permit. The average number of analyses possible per day is included in each method outline; this number could only be increased by reassigning staff from other analytical functions. This number provides a guideline which will allow samplers to regulate their sample inputs and avoid pre-empting a large portion of the daily capacity for any given test. Your co-operation will assist the laboratories in maintaining a quick and efficient analytical service.

Sampling information is provided in each method outline. If there are any questions, the sampler should consult with laboratory staff before the samples are collected. For more information on sampling techniques, the reader is referred to the "Sampling Guide".

Sample Containers and Preservation Techniques

For the protection of Laboratory Staff, it is imperative that information about hazardous substances in the sample be clearly written on the sample container. A proper warning label should be placed on the bottle (toxic, explosive, corrosive, etc).

In general, water samples are collected in 500 mL plastic jars for inorganic tests and in 1 L glass bottles or 250 mL glass jars for organic tests. Many procedures call for sample preservation. The individual outlines indicate both the preferred container type as well as the preservation techniques for each parameter. While some parameters are stabilized by a certain preservation technique others may be destroyed or altered. It is the responsibility of the sampler to obtain the proper sample container and to use the proper preservation technique. For additional detail refer to the Sampling Guide. Pet (polyethylene terephthalate) plastic containers, recommended in this report may not be available immediately. In the interim use containers recommended in the Sampling Guide.

For domestic water, surface water and precipitation samples, heavy metals with the exception of mercury and hexavalent chromium, are preserved by adding 20 drops (1 mL) of concentrated nitric acid to one liter of sample after the sample is in the bottle. **Never add acid to an empty plastic container prior to sampling** as strong acid will activate plastic surfaces in such a way that some heavy metals may be adsorbed onto the container walls. Foil lined caps must be avoided when sampling for trace quantities of heavy metals.

Aqueous samples for organic analyses should be submitted in 1 L glass bottles with Teflon or foil lined caps. The sample should be refrigerated but not frozen.

PCB and pesticide samples must be collected in baked 1 L brown glass bottles with Teflon lined lids or **30 mL vials for concentrated samples**.

Sludge samples should be collected in wide mouth jars and never filled more than two thirds. The extra space is required as an expansion zone for gaseous products that may be formed. Failure to submit samples in this manner may result in container explosion in transit or at the laboratory. **Overfilled sludge samples are discarded without analysis.** Sludge samples for trace organic analyses must have a foil lined cap.

Vegetation samples should be placed in perforated polyethylene bags and transferred to refrigerated storage as quickly as possible.

Any clean glass or plastic container is acceptable for inorganic analyses of sediment, soil or biomaterial samples. The pomade jar and twirl-top bags are the most commonly used. For short term preservation, refrigeration at 4°C is recommended. Drying, as a preservation technique is recommended except for samples requiring analysis of potentially volatile parameters. If drying in the field is not practical, the laboratory should be notified so that the samples can be dried without delay.

In many instances, generally arising from citizens' complaints, it becomes necessary for field personnel to collect samples for constituent identification by means of microscopic, X-ray diffraction, electron probe and other techniques. The types of material normally encountered are visible solids present in air or water, that are a cause of nuisance or concern to the complainant. To facilitate the collection of these samples, a sampling kit is available from the Laboratory Services Branch for each district office in the province. Supervisory personnel should obtain and maintain this kit. It contains detailed sampling instructions.

A general summary of sampling containers available from Central Stores is listed in **Table 2**. Since containers are continually being replaced by improved designs and materials, those approved previously in the "Sampling Guide" will be accepted for several months until old stocks are exhausted.

Sample Volumes

Table 3 specifies the volume of sample required for proper analysis of all sample types. For a sample containing low concentrations of a substance, the largest practical volume is tested in order to provide a sufficient quantity of that substance for reliable detection. Conversely, submission of large quantities of concentrated chemicals is unnecessary and strongly discouraged because of safety, handling, and disposal problems.

The sampler is expected to calculate the total volume of sample required by summing the individual volumes for all the analyses requested and to submit the appropriate quantity. A number of tests may be determined simultaneously on the same aliquot.

1. Both total phosphorus and total Kjeldahl nitrogen can be determined on a single 75 mL aliquot.

2. Nitrate-, nitrite- and ammonia-nitrogen and filtered reactive phosphorus can all be determined on a single 75 mL aliquot.

3. Both sodium and potassium (or calcium and magnesium) can be determined on a single 40 (or 75) mL aliquot.

4. Aluminum, copper, cadmium, cobalt, chromium, iron, lithium, molybdenum, manganese, nickel, silver, strontium, zinc, tin, barium, beryllium, gold, lead, thallium, vanadium and titanium, can all be analyzed on a single 500 mL aliquot if necessary.

5. Dissolved organic and dissolved inorganic carbon can be determined on the same 50 mL aliquot.

Samplers are asked to submit at least 180 mL of sample in excess of their original total estimate to allow for rinsing of tubes and possible repeat analyses. Failure to submit sufficient sample volume will result in "Insufficient Sample" being marked on analysis report sheets. The sampler must also use the proper containers.

In certain cases, where the sampler is unable to obtain sufficient sample volume or when resampling is impossible, analysis may still be performed if special techniques are used in the laboratory. This can only be achieved after consultation with laboratory staff before sample submission.

Sample Submission

Sample Bottles - All sample bottles must be clearly labelled and contain the following information (keep in mind that the analysts may never see the submission sheets, - only the label):

1. Proper cautionary notes to analysts, if **hazardous** substances are present (cyanide, strong acids or bases, PCB's etc.).

2. A sample (sender's) number.

3. Some other identification, normally the sample source or type (e.g. "Lake Temagami - Sharp Rock Inlet").

4. Presence of any chemical preservative added; all others will be kept refrigerated or frozen until time of analysis.

5. Indication of the specific analyses to be performed on that sample bottle (preferably the same codes used on submission sheets - ie. PHNOL for phenolics).

Submission Forms - Submission and Request for Analysis forms MUST accompany all samples and must be completed as described in the "Sampling Guide".

Submission Form-

- the first and last field sample numbers
- the sample program code
- the laboratory to which the samples are sent
- the priority of the submission, normal or high (if high, a phone call must precede and authorization forms must accompany the submission - see Table 4)
- the sampling agency code
- the date the submission was sent to the laboratory (Day/Month/Year)
- the name of the sampler
- the phone number of the sampler
- the client code of the person or group responsible for the submission or the name, address, phone number and establishment of the person to whom the report should be sent
- the client codes or names and addresses of other people to whom reports should be sent

Request for Analysis Form-

- the field sample number
- the sample type code
- the number of containers sent
- the sampling date (day/month/year) and time
- the sample location or station
- a sample description such as soil, sewage, etc.
- the test group(s) requested
- any deletions from the test group, on the same line as the test group
- any individual test additions

If a group of related samples is submitted, a map of the area or the relative location of waste inputs is very helpful to the laboratory staff. Please number the samples in a logical order, e.g. downstream in a river. All analyses are routinely screened for anomalous results before they are released. Suspicious results might be improperly rejected without details of the location to confirm their validity.

Analytical Constraints

When a sample is sent to the laboratory a description of known unusual or hazardous constituents should be included on container labels and on the submission sheet. Interferences (reactions which produce false analytical results) can often be eliminated by pretreatment if the analyst is forewarned. **Samples known to contain cyanide, arsenic, PCB's or other toxic materials should be clearly marked for the protection of laboratory personnel.**

Identification of unknown contaminants is very time consuming. Samples should be as large as possible to allow a wide range of exploratory tests. The sampler should indicate whether qualitative or quantitative results are required. Any available information concerning the sampling point, possible contaminants, and industries implicated is extremely important for such samples.

Samples which are not homogeneous present analytical difficulties because it is virtually impossible to take a representative aliquot. If the sampler is only interested in one phase (aqueous, solid or immiscible organic), he should label the submission form accordingly. Otherwise the laboratory will consider the whole sample, and take aliquots of the mixture (an exception follows).

Erroneous results for trace heavy metals and other inorganic constituents in water will be obtained if particulate material is inadvertently present in the sample, e.g. for samples of groundwater in landfill monitoring wells. Great care must be taken to minimize such contamination. Field filtration may be necessary prior to addition of acid preservative. However, in some cases field filtration is not feasible and an apparently clear sample may form precipitates several hours or days after collection.

To avoid these problems the Water Quality Section has developed a protocol for all landfill samples **(other types of samples must be included in separate submissions or they will be treated as landfill samples).** All samples in a landfill submission will get equivalent treatment (LE type only): pH, conductivity, and alkalinity will be determined immediately on the supernatant or filtered sample; the supernatant is then diluted and analyzed, if conductivity is high (>1500 µS/cm), or analyzed directly at designated workstations. **BOD, turbidity, and suspended solids analyses will not be performed.** Note that total Kjeldahl nitrogen and total phosphorus performed on the supernatant describe only that portion of the sample.

The "routine" analytical procedures are a compromise designed to provide adequate sensitivity for most sample types while still maintaining acceptable throughput and turnaround. Detection limits of these procedures are matrix dependent. Generally the more extraneous material present, either organic or inorganic, the poorer the detection limit and the longer the analysis time. The material may interfere and/or contaminate both the preparative and analytical phases of a procedure. For example it is more difficult to measure cadmium accurately at 100 ppb in a sediment than 0.2 ppb in surface water. As a rule of thumb:

Matrix	**Relative Detection Limit**
Pristine waters	1
Ground waters (not landfill leachate)	5
Sewage, industrial waste, landfill leachate	10-100
Solids	100-1000

Collection of Samples

Sterile sampling bottles are available from Central Stores in Toronto. For special studies, alternate bottles are obtainable directly from the Microbiology Section. It is the responsibility of the sampler to use aseptic techniques when handling these sterile bottles. Inaccurate results are obtained when contamination occurs through faulty handling of sterile containers.

Surface Samples

Clamp the bottle onto a sampling pole before removing the cap. Touch only the outer surface of the cap when opening the bottle. The inner lip and liner must not come in contact with anything except the atmosphere. If the inner surfaces of the cap or bottle lip are accidentally touched, the sample has been contaminated and should not be submitted. It is a recommended procedure to hold the cap with the fingertips until the sample has been taken. The cap must not be set down while the sample is being taken as this will result in contamination.

Surface sampling is accomplished by quickly lowering an inverted sample bottle below the surface of the water, turning it upright and holding it there until all bubbles have stopped coming to the surface. When sampling a stream or river, place the bottle with the mouth facing into the current. When sampling near shore, care should be taken to get a sample which has not been contaminated with sediment. The bottle is then removed from the water, the level adjusted to the top of the label, immediately capped, and then unclamped from the sampling pole. Samples must be collected using this prescribed technique. The use of a dipper or other sampling device will result in contamination.

Depth Samples

Depth samples are taken using sterile sampling bulbs which can be obtained from the Microbiology Section in Toronto. Bulbs should be used as quickly as possible. If not used, they should be returned to the Microbiology Section within a maximum of two weeks, otherwise the rubber will crack and the bulb will not open. The same care that is used with sampling bottles must be used in the handling of bulbs. The glass plugs supplied have been sterilized within a cellophane envelope and must not come in contact with any contaminated surfaces when they are being removed from the cellophane envelope. If, for some reason, the sampler should run out of glass rods, he may dip the metal plug into alcohol and flame it. After flaming, the plug is immediately inserted into the bulb, taking the usual precautions when handling sterile equipment. The use of the metal plug is discouraged and it should only be used in rare instances when the sample could not possibly be obtained at a later time in the correct manner.

Tap Samples

Prior to sampling from a tap, the water should be allowed to run at full flow for approximately two minutes. The strong flow will clean out residual contamination around the orifice of the tap thus providing for a more representative sample. The water pressure may then be reduced to permit taking the sample without excessive splashing which could result in contamination of the sample.

Fill the bottle to the top of the label, being certain that the mouth of the bottle does not come in contact with the tap or any contaminated surface. The cap must also be handled aseptically as described previously.

Perishability

While being transported to the laboratory, samples undergo changes (increases or decreases) in bacterial population, depending upon the initial bacterial density, the presence of growth-promoting or toxic substances in the water and the environmental conditions under which the samples are held. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 24 hours.

Drinking water, waste and river samples that require transportation by public carriers received 24 to 48 hours after collection can be analyzed. Samples received after three days are rejected. Negative coliform results for drinking water samples arriving more than 48 hours after collection should be considered incorrect and fresh samples should be collected.

Sample Containers and Preservation Techniques

Presterilized 250 mL bottles with red labels usually provide adequate volume for the routine analysis of water from distribution systems, surface waters and well waters. It is particularly important that chlorinated waters, or waters in which a chlorine residual is suspected, be sampled in bottles containing sodium thiosulphate as a preservative (red label). Sodium thiosulphate will neutralize the disinfecting capability of the chlorine thereby preserving the existing microbial population at the time of sampling. Before samples are collected for sulphur oxidizing bacterial analyses, the Microbiology Section should be contacted to determine whether thiosulphate should be used or not.

All samples should be collected early in the week and shipped to the laboratory without delay. During spring, summer and fall, samples must be packed in ice to minimize biological activity. During winter, samples should be packed in an insulated container that will prevent freezing while still keeping them cold.

Sample Volumes

In general, one bottle or bulb per sample provides sufficient volume for standard analysis. If, however, the bacterial levels expected are very low or extra parameters are being requested, then additional samples may be required. Consultation with the Microbiology Section is advisable in such cases. When a duplicate sample is being taken, it should be obtained at the same time as the first sample. This can be achieved for surface samples by clamping two bottles on a sampling pole and for depth samples by placing two bulb samplers on the sampling line in a 'piggyback' fashion.

For sample submission of microbiology samples, refer to the section on sample submission.

SHIPPING PROCEDURES

Couriers provide the fastest and most reliable service for the shipment of environmental water samples in Ontario. Air Express, parcel post, bus companies and other services discourage the shipment of water samples because of the damage caused to other shipments when breakage occurs.

Contract numbers are important as they provide the only means for tracing a lost shipment. Every shipment is assigned a contract number, but it is generally up to the sampler to attach this contract number to each carton of his shipment. Samplers are urged to keep a record of all their contract numbers.

The following addresses should be used when shipping samples to the various laboratorires.

1. **Main Toronto Laboratory**

Ontario Ministry of the Environment,
Central Stores,
125 Resources Road,
Highway 401 and Islington,
Rexdale, Ontario.

2. **Southwestern Region - London Laboratory**

Ontario Ministry of the Environment,
Southwestern Regional Laboratory,
985 Adelaide St. S.
London, Ontario.

3. **Northwestern Region - Thunder Bay Laboratory**

Ontario Ministry of the Environment,
Thunder Bay Regional Laboratory,
421 James St. South
Thunder Bay, "F", Ontario.

4. **Southeastern Region - Kingston Laboratory**

Ontario Ministry of the Environment,
Southeastern Regional Laboratory,
133 Dalton St.,
Kingston, Ontario.

Sample Enquires

All samples received by the Laboratory Branch are assigned alpha-numeric codes according to sample type. When the analyses are completed, the results are entered onto the report sheets, checked by the scientist or technician in charge, and sent to the people listed on the sample submission form. All original submission sheets are retained in the sample reception files. Laboratory staff are prepared to answer questions regarding the receipt and progress of samples but require at least the following information:

- the submission number if known.
- the name or numerical code of the sampling program.
- the date the sample was submitted to the laboratory.
- the municipality from which the sample was taken.
- the sampler's name.
- sample identification number including laboratory number if known.
- the sample type (water, sewage, Great Lakes, etc.).
- the tests requested.

LIMITS OF CONCERN FOR PARAMETERS

Environmental, ecological or health limits are mentioned for many of the parameters in this report. These should not be used as primary references, but rather as indicators of potentially undesirable levels.

Definitions of LC-50 and LD-50 can be found in the Aquatic Toxicity outline.

ACIDITY

The acidity of a water is a measure of its capacity to neutralize a base or to donate protons.

The major component of acidity in natural waters is carbonic acid which is formed by the absorption of carbon dioxide. Other lesser components include: portions of other weak acids such as phenolics or undissociated tannic acid; hydrolyzable metallic salts, particularly iron and aluminum; and mineral acids which cause an unusually low pH. Acidity is a capacity function and can only be interpreted in terms of specific substances when the chemical composition of the sample is known. Carbonic acid acidity is the predominant form between pH 4.5 and 8.3. Below pH 4.5, mineral acidity predominates.

The acidity of natural waters is increased by industrial activity. Drainage from mine tailings may contain sulphuric acid and hydrolyzable iron due to the aerobic oxidation of pyrite by sulphur bacteria. Oxides of sulphur and nitrogen emitted to the atmosphere from industrial sources contribute to acidic precipitation.

Acid waters inhibit biological activity and have objectionable corrosive properties.

Sampling

Samples should be collected in PET plastic containers. Bottles should be filled completely so that no air bubbles remain. Samples should be refrigerated but not frozen and transported to the laboratory as soon as possible. Soil samples should be collected in pomade jars.

Method

Samples are titrated automatically with a standardized sodium hydroxide solution. Total Fixed Endpoint Acidity (TFE) is calculated from the volume of titrant used to pH endpoint of 8.3. Gran Acidity is calculated by subjecting the titration data to Gran's plot.

Any sample which can be titrated by a base can be analysed; including precipitation, surface water, domestic water and industrial waste. However, the laboratory should be notified if acidity is required on liquid industrial waste, since the equipment was designed for "clean" samples. Samples suspected of having high buffering capacity can be diluted prior to analysis.

Time of Analysis

Approximately 70 automatic acidity titrations can be performed per day. Time required to perform titrations is directly related to acidity.

ALKALINITY

The alkalinity of water is a measure of its capacity to neutralize acid. Bicarbonate and carbonate ions are generally the major contributors to the alkalinity of natural water but any ion entering into a neutralization reaction with strong acid may contribute to the alkalinity as long as the reaction takes place above the pH of the specified endpoint. The hydrolyzable equivalents of silicates, phosphates, arsenates, aluminates and cyanides may all contribute. Hydroxides, which may be present in water softened by the lime or lime-soda process or previously in contact with cement, may also contribute to the alkalinity.

The alkalinity of water is generally used to assess the buffering capacity or the water's ability to resist a change in pH. This means that if an acidic waste is discharged into a natural water system, the effect on the water may not necessarily be a decrease in the pH, but may be detected as a drop in the alkalinity.

A total alkalinity represents the amount of acid required to fully neutralize all acid reactive substances in the sample. The most precise estimate of total alkalinity is obtained by Gran's analysis of the titration curve. The titration of a sample to a fixed endpoint of pH 4.5 yields a less precise estimate, particularly with poorly buffered samples.

Waters with a total alkalinity below 15 mg/L of calcium carbonate are considered to be very poorly buffered.

Sampling

Samples should be collected in PET plastic containers. Bottles should be filled so that no air bubbles remain. Samples should be refrigerated but not frozen. Immediate delivery to the laboratory is essential. Soil samples should be collected in pomade jars.

Method

Samples are titrated automatically with a standardized sulphuric acid solution. Low level river and precipitation samples are subjected to Gran analyses of the titration data to estimate the total alkalinity. Particulates are not included in the aliquot taken for analysis. A fixed endpoint alkalinity (pH4.5) is determined for domestic waters, high buffering surface waters, sewage, leachates, and industrial wastes.

Time of Analysis

Approximately 150 surface water and domestic water samples each may be analyzed per day.

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ALUMINUM

Aluminum is one of the most abundant elements in nature and is widely distributed in both soluble and insoluble forms. The native metal and its oxide are insoluble, but many salts are highly soluble. Its most important sources include bauxite, cryolite, alum, corundum and kaolin minerals. Aluminosilicate minerals are abundant in igneous and sedimentary rocks and soils. As the minerals weather, aluminum may be left as an insoluble residue such as bauxite. Aluminum compounds occur naturally in water, but may also be derived from industrial wastes or effluents from water treatment plants where alum is commonly used as a precipitating or flocculating agent. Aluminum concentrations are generally less than 1.0 mg/L; however, acid mine drainage may lead to increased levels in certain areas. While aluminum is amphoteric, it is seldom found in the anionic form.

Aluminum is used extensively in domestic utensils, wire, exterior building finishes and in any industrial applications where a strong, light, easily constructed material is needed. Aluminum is also used in dye and paper manufacturing and in the tanning industry. The presence of small amounts of aluminum in water may interfere with certain industrial processes.

Oral toxicity of aluminum and aluminum salts seems to be negligible although ingestion of large amounts seems to alter the body's method of inorganic phosphate elimination. Inhalation of the element or its salts may cause lung problems. Toxic effects on plants have been reported for extreme conditions at both ends of the pH scale, but at a neutral pH, toxicity effects are negligible. Since aluminum has not been shown to be harmful to human health, no drinking water guidelines have been proposed. A limit of 0.100 mg/L has been proposed to protect aquatic biota. The Water Quality Criterion for livestock watering is 5.0 mg/L.

source?

Sampling

PET plastic containers are required for domestic and industrial waste samples. 20 drops of HNO_3 per liter of sample are required as a preservative. Soil and sediment samples should be collected in pomade jars. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Aqueous samples may be preconcentrated before digestion with mineral acids. Solid samples are also digested with mineral acids. Sewage, sludge and Hi-Vol filters are analyzed using Inductively Coupled Plasma (ICP) atomic emission. All other matrices are analyzed using atomic absorption.

Time of Analysis

The time of analysis depends on the amount of preparation required. Approximately 50 aluminum determinations may be performed per day for aqueous samples.

ANTIMONY

Antimony is not generally found in the native state. Its major forms in the natural environment are as insoluble sulphides such as the mineral stibnite and oxides such as the minerals cervantite and valentinite.

Antimony is generally found in only trace amounts in natural waters as any dissolved antimony is rapidly adsorbed onto suspended material or precipitated from solution as an oxide. The major sources of antimony in waters are mine wastes, weathering of rock and industrial effluents.

Pure antimony metal is a poor conductor of heat and electricity and is used in the semiconductor industry. The most important use of antimony is as an alloying ingredient for imparting hardness and stiffness to other metals. Batteries, anti-friction alloys, metal used for typesetting and cable sheathing use a large proportion of the metal produced. Paints, ceramic tile, glass, rubber and pottery are other industries using antimony.

Antimony and several of its compounds are known to be toxic to man. It accumulates in the liver and is detrimental to the heart. Drinking water objectives have not been set. Exposure in air should be limited to 0.5 mg/m^3 based on a 40 hour work week. The 96 hour LC 50 for fish ranges from 9 to 80 mg/L, depending on the water hardness.

Sampling

Aqueous samples are collected in PET plastic jars and preserved with 20 drops of HNO_3 per liter of sample. Soil and sediment samples should be collected in pomade jars. Vegetation samples are collected in perforated plastic bags and must be refrigerated.

Method

The sample is digested with an oxidizing acid mixture. Stibine gas is generated in acid solution by sodium borohydride, atomized in a heated quartz tube and measured by atomic absorption.

Time of Analysis

Approximately 50 antimony analyses can be performed per day.

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ARSENIC

Arsenic is commonly found in nature as the minerals realgar, orpiment, arsenolite and arsenopyrite. The two most common states are As(III) and As(V). Arsenic(V), as arsenate (AsO_4^{3-}) is the stable form in aerobic waters. Arsenite may be present if oxidation to As(V) is incomplete. Arsenic (III) will be the predominant form under anaerobic conditions. In Canada, the dissolved arsenic concentration in river samples collected between 1972 and 1977 ranged from 0.001 to 0.050 mg/L. Arsenic concentration in natural waters varies from less than 0.005 to 0.620 mg/L, Great Lakes from less than 0.0001 to 0.0012 mg/L, sediments from 0.5 to 14.0 mg/kg and fish from 0.03 to 0.12 mg/kg.

Arsenic may be present in natural waters by dissociation of minerals, industrial discharges and pesticide applications. Common industrial applications include the processing of metal ores, glassware, ceramics and tannery products. Manufactured products containing arsenic include wood preservatives, herbicides and insecticides.

As a pollutant in water, arsenic is commonly found as arsenite and arsenate, as the gas arsine, and as organic arsenicals such as methylarsenate, phenylarsenate and trimethyl arsine.

No!

The element and its inorganic salts are highly toxic to humans and are known to exhibit carcinogenic properties. Since arsenic is eliminated from the body very slowly, it tends to accumulate, concentrating in the hair, skin and nails. Arsenic should not be present in domestic waters at concentrations higher than 0.01 mg/L of As. Levels should not exceed 0.1 mg/L to protect aquatic life. 0.05 drinking water

Sampling

PET plastic containers are used for water, sewage, industrial waste and sludge samples. 20 drops of HNO_3 per litre of sample are required as preservative. Vegetation samples may be collected in perforated plastic bags and refrigerated. Soil and sediment samples may be collected in pomade jars.

Method

Sample aliquots are digested with an oxidizing acid mixture. Arsine gas is generated in acid solution by sodium borohydride, atomized in a heated quartz tube and measured by atomic absorption. High concentrations of Se, Sb, Sn, Co, Ni, Cu, Mo and Pt may give erroneous results.

Time of Analysis

Approximately 50 arsenic analyses can be performed per day.

ASBESTOS

Asbestos is a generic term for several fibrous silicate minerals of the serpentine and amphibole groups. Chrysotile or white asbestos is the sole species classified in the serpentine group and is by far the most abundant kind of asbestos. The amphibole group includes asbestiform types, namely crocidolite, amosite, anthophyllite, tremolite and actinolite.

Asbestos occurs as an environmental water and air pollutant through both natural causes and human operations. Although erosion of rock formations introduces fibres into bodies of water, modern usage of asbestos in over four thousand industrial and domestic applications has provoked concern over the amount introduced into the environment by the mining and milling of asbestos and the manufacture and use of asbestos products.

Growing concern over the long-term health effects of airborne asbestos has led to the belief that asbestos concentrations in the environment should be carefully monitored.

Sampling

A Hi-Vol sampler, equipped with a transducer and continuous air flow recorder, is fitted with a Nuclepore filter of dimensions 20 x 25 cm and 0.5 μm pore size. Due to the difficulty in handling the Nuclepore filter under field conditions, installation of the filter in a Hi-Vol cassette or filter holder is carried out in a sheltered environment. The cassette may then be attached to and removed from the Hi-Vol at the given field location. After removal of the filter from the cassette, the filter is placed on a separator sheet (supplied with the filter), with the sample side facing up and both are folded in half along the 20 cm width. The entire package is then placed in a glassine envelope and delivered to the laboratory together with the air flow data.

For water samples, a polyethylene bottle of 1 liter capacity is rinsed at least three times with the water from which the sample is to be taken and then filled to one inch from the top of the bottle with the water sample. Samples must be shipped to the laboratory without delay and be protected from freezing.

Bulk samples may be submitted in plastic bags, plastic dishes or glass jars.

Method

The particulate material from a representative aliquot of the air or water sample is deposited on a 200 mesh electron-microscope grid which is examined at 20000X magnification. Fibres are classified, counted and sized and the fibre and mass concentrations of chrysotile and amphiboles are calculated and reported.

Bulk samples are analyzed by polarized light microscopy. The percentages of chrysotile, amosite and crocidolite are reported.

Time of Analysis

Each sample requires from 3 to 8 hours or more depending on the total particulate loading of the EM grid.

BARIUM

Barium is commonly found in nature as the minerals barite (barium sulphate) and witherite (barium carbonate). It is also found in igneous rock, primarily feldspar, where it is known to replace potassium. Barium ions in solution are rapidly precipitated by sulphate and carbonate. Weathering and industrial discharges are the main sources of barium in natural waters. Industrial application of barium includes the manufacture of metal alloys, paints, pigments, paper, soap, rubber, linoleum, flares, cement and various other products.

Insoluble barium salts are non-toxic. Barium sulphate, for example, is used as a contrast material in the radiological diagnosis of ulcers. Soluble salts of barium, however, are readily absorbed through the skin or gastrointestinal tract and exhibit highly toxic effects on the nervous system if present in sufficient quantity. Symptoms include violent muscular spasms, paralysis and cardiac arrest. Trace quantities of soluble barium are known to accumulate in bones, but are not believed to exhibit a cumulative toxic effect like lead or cadmium. The maximum acceptable concentration of barium in a domestic water supply is 1.0 mg/L. There are currently no guidelines for livestock or irrigation waters. Levels of 1.0 mg/L are considered hazardous for marine organisms. The 96 hour LC 50 levels for invertebrates and fish are 10 - 15 mg/L and 50 - 100 mg/L respectively. Aquatic organisms may concentrate barium in water by a factor of about 150.

Sampling

PET plastic jars are required for aqueous samples. Add 20 drops of HNO_3 per liter of sample. Pomade jars should be used for soil and sediment samples. Vegetation samples should be placed in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested with the appropriate acid mixture and analysis is accomplished by Inductively Coupled Plasma (ICP) atomic emission spectrophotometry.

Time of Analysis

Approximately 50 barium analyses may be completed per day.

BERYLLIUM

Beryllium is a trace element which does not usually occur in natural waters. It is present primarily in particulate form rather than in dissolved form in all except very acidic conditions. Beryllium chloride, nitrate and sulphate are soluble in water but beryllium carbonate and hydroxide are insoluble. Levels seldom exceed 0.001 mg/L.

Weathering of minerals such as beryl accounts for some of the beryllium in natural waters but the majority comes from industrial discharge.

Beryllium metal has a high strength to weight ratio, a high melting point, high modulus of elasticity, high thermal conductivity and is quite resistant to corrosion. Beryllium copper alloys show many of the same properties. As a result, the metal and its alloys have found widespread use in the automotive, aircraft, spacecraft, missile and electrical industries. Beryllium is also used in X-ray lithography for reproduction of micro-miniature integrated circuits.

Beryllium and its salts are toxic and should be handled with the greatest of care. Exposure to airborne beryllium dust should be limited to 2 $\mu g/m^3$ over a 40-hour work week. The major toxic hazard potential comes from inhaling beryllium containing fumes and dusts. As yet, there are no drinking water guidelines.

At low levels, beryllium is toxic to plants, since it inhibits photosynthesis. Therefore irrigation waters used continuously should not contain concentrations greater than 0.10 mg/L. Levels of 0.011 mg/L may pose a threat to aquatic organisms in waters with a hardness of less than 75 mg/L of $CaCO_3$.

Sampling

PET plastic jars are required for aqueous samples. Add 20 drops of HNO_3 per liter of samples and sediment. Soil samples should be collected in pomade jars while vegetation samples should be collected in plastic bags and kept refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

The sample is digested with an appropriate acid mixture and analyzed by atomic absorption.

Time of Analysis

Approximately 50 beryllium analyses can be performed per day.

BIOCHEMICAL OXYGEN DEMAND (BOD_5)

The BOD_5 test is a measure of the dissolved oxygen required for the chemical and the aerobic bacterial oxidation of carbonaceous and nitrogenous substances in a five day period at 20°C in the absence of light.

The most frequent damage caused by the discharge of organic wastes to natural waters, is the reduction of dissolved oxygen concentrations to levels which cannot support normal aquatic life. This oxygen depletion arises via the bacterial oxidation of carbon and nitrogen materials and eventually leads to a general deterioration of water quality for all uses. Ideally the BOD_5 should be supplemented by on-site dissolved oxygen measurements in order to assess the effects of stream conditions which offset or accentuate the utilization of dissolved oxygen. The five day period for analysis is arbitrary but has become a standard. Longer periods which include daily oxygen demand measurements are used to measure rates of oxidation and the ultimate demand (k-rates).

The precision of the method is seriously reduced if the sample contains large fragments of suspended solids or an immiscible layer of oil. Representative portions of such samples cannot be properly aliquoted and the results are not generally reproducible within acceptable limits. Biocides such as trace metals and toxic organics seriously interfere with the BOD test and industrial wastes such as tailing effluents and landfill leachates should not be analyzed for BOD.

Sampling

The BOD_5 is a perishable quantity since it is readily influenced by bacterial activity. Immediate delivery to the laboratory is essential (preferably before Friday afternoon). Samples should be collected in PET plastic containers and kept refrigerated.

Method

Dilutions are prepared on the original sample as necessary, and the dissolved oxygen depletion is measured after a five day incubation period in a sealed bottled at 20°C. Only those dilutions which give a dissolved oxygen depletion between 25% and 75% are accepted for calculating the BOD_5. Dissolved oxygen levels are measured using a membrane electrode which is calibrated using a reference table for oxygen saturated water, corrected for temperature and barometric pressure. Calibration is checked by the Winkler Method. Samples with high chlorine residuals and adverse pH levels are chemically adjusted to remove these toxic influences and are then seeded with sewage. A technique which suppresses the nitrogen-oxygen demand is performed upon special request.

Time of Analysis

By definition, the BOD_5 test will take five days to complete. However, approximately 200 BOD_5 analyses per day may be set up on Thursday and Friday.

BORON

Boron occurs in nature as the minerals tourmaline, borax, colemanite, boronatrocalcite and boracite. These mineral deposits may significantly increase the boron concentration in natural waters. Granites, pegmatites, gabbros and serpentine rocks may also contribute significant amounts of boron by weathering. Boron in water can exist as undissociated boric acid, borate ion and as various polyanionic species. Boron can also enter the waterways through cleaning compounds and industrial waste effluents. Surface water concentrations average about 0.1 mg/L but concentrations may be much higher if groundwater inputs make up a large portion of that water.

Boron is used as a neutron absorber and shielding material in nuclear power plants, in the manufacture of glass, porcelain enamels, detergents, pharmaceuticals, abrasives and dyes. It is also used in fireproofing and waterproofing materials as well as in bactericides, herbicides and fungicides.

Boron is an essential plant nutrient at low levels but high levels may have harmful effects. The maximum permissible concentration of Boron in irrigation waters is 0.75 mg/L. Boron does not appear to accumulate to any extent in the human body. Large doses may cause digestive problems and affect the nervous system. The maximum allowed concentration in irrigation waters is 5.0 mg/L. Levels above 5.0 mg/L may constitute hazard to some marine organisms.

Sampling

PET plastic containers should be used since boron is a major component of glass. 20 drops of HNO_3 per litre of sample should be used as a preservative. Vegetation should be collected in plastic bags and refrigerated. Fish samples should placed in plastic bags and frozen.

Method

Boron in terrestrial and vegetation samples is determined colorimetrically using Azomethine H or by atomic spectrometry techniques. A preliminary Chemical treatment of some sort is generally performed depending on the sample matrix. Hot water extraction is sometimes used for soils and sediments. High concentrations of humic material, iron, aluminum, zinc and copper may interfere with the Azomethine H method.

Time of Analysis

Approximately 20 boron determinations may be performed per day.

CADMIUM

Cadmium levels in natural waters are generally below 0.01 mg/L unless there is an industrial source nearby. While weathering of rocks and minerals such as greenockite (cadmium sulphide) adds some cadmium to waters the principal sources are industrial processes. Since greenockite is often found with zinc sulphide ore, the mining and smelting of zinc, lead and copper release large amounts of cadmium to the air and water. It may be discharged from the manufacturing of nickel cadmium batteries, various alloys, solders, ceramics, pigments and printing inks. It is also used in some pesticides and fertilizers. The burning of fossil fuels accounts for a large part of the cadmium emissions.

All forms of cadmium are highly toxic, and once ingested are likely to remain in the body for a long time, becoming concentrated in the liver, kidneys and other organs. Cases have been reported where toxic effects did not become apparent until many years after the last exposure. The water quality criteria for cadmium in domestic water supplies is 0.01 mg/L. The presence of cadmium reduces plant growth. Since it can be accumulated within plants which may be used as a food source, irrigation waters should not contain more than 0.050 mg/L when used on neutral or alkaline soils and 0.010 mg/L when used continuously on all soils. Levels above 0.0002 mg/L may be harmful to some aquatic organisms.

Sampling

When levels below the normal range of application are required, a notation must be included on the sample submission form so that the sample can be preconcentrated prior to analysis. 500 mLs is required exclusively for the preconcentration process.

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per litre of sample. Use pomade jars for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested with an appropriate acid mixture and then analyzed. Sewage, sludge and Hi-Vol samples are analyzed by Inductively Coupled Plasma (ICP) atomic emission spectrophotometry. All other matrices are analyzed by atomic absorption spectophotometry. Polarographic techniques may be used for ultra trace concentrations in some matrices.

Time of Analysis

Approximately 50 analyses may be performed per day.

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CALCIUM

Calcium ranks fifth in the order of natural elemental abundance and is commonly found in deposits of limestone, dolomite, gypsum and gypsiferous shale. It is generally present in all natural waters at levels which depend on the amount of contact with specific geological formations. Typically, levels in waters are less than 15 mg/L but waters in contact with carbonate rocks may contain up to 100 mg/L. For this reason, surface waters generally tend to have lower calcium levels than ground waters. The calcium content may vary from zero to several hundred milligrams per liter depending on the source and treatment of the water.

Calcium enters the waterway through leaching of soils, weathering of rocks, effluents of breweries and glue factories, and from road salt.

Calcium is an essential element for all life forms. It enhances biological productivity in plants and promotes proper bone and teeth formation in animals. Aqueous calcium compounds are generally non-toxic and represent no known health hazard.

While low concentrations of calcium assist in preventing the corrosion of metal pipes by forming a protective layer of calcium carbonate, high concentrations cause excessive scale which lead to restricted flow and pressure build-ups. Calcium is the major contributor to water "hardness".

A limit of 200 mg/L has been proposed for drinking water with an objective of 75 mg/L. source?

Sampling

Aqueous samples should be collected in PET plastic containers. Non- routine industrial wastes, or any other samples where high levels of heavy metals are suspected, should be preserved with 20 drops of nitric acid per liter. Sludges need no preservation. Soils should be collected in pomade jars. Vegetation samples should be collected in perforated plastic bags and kept refrigerated.

Method

Most samples including domestic water, precipitation, river, industrial waste, soil and dustfall samples are analyzed by atomic absorption. Sewage and sludge are analyzed by Inductively Coupled Plasma (ICP) atomic emission. For calcium analyses in vegetation by X- ray fluorescence, see Matrix Elements in Vegetation.

Time of Analysis

Approximately 200 analyses can be performed per day using atomic absorption. Approximately 50 samples per day can be performed by Inductively Coupled Plasma (ICP).

The dissolved inorganic carbon test measures dissolved carbon dioxide, bicarbonate and carbonate. Inorganic carbon enters the water from the atmosphere and by weathering of carbonaceous rocks such as limestones and dolomites. Bicarbonate salts may also be added by industrial effluents.

Carbonate equilibria in natural water systems are governed by the equations below, where pH, the partial pressure of CO_2 and sample alkalinity are the controlling factors.

$$CO_2\ (g) + H_2O\ (l) = H_2CO_3\ (aq)$$

$$H_2CO_3\ (aq) + OH^-\ (aq) = HCO_3^-\ (aq) + H_2O\ (l)$$

$$HCO_3^-\ (aq) + OH^-\ (aq) = CO_3^{2-}\ (aq) + H_2O\ (l)$$

Generally, below pH 4.5, H_2CO_3 (aq) is the predominant species, between pH 4.5 and 8.3, bicarbonate is the major species and above pH 8.3, carbonate is the major ion present.

Dissolved inorganic carbon is another measure of the buffering capacity or alkalinity of a natural water and there is a relationship between DIC and alkalinity.

The dissolved inorganic carbon test can be used as one of the factors to estimate primary productivity levels by determining how much dissolved inorganic carbon is available for uptake by algae for photosynthesis.

At present there are no established limits for carbonate or bicarbonate in waters, however high levels may be objectionable due to precipitation with calcium and magnesium to form a scale on metal surfaces.

Sampling

Typical surface and ground water samples should be collected in PET plastic containers. Samples for the low dissolved inorganic carbon test should be collected in hard glass (Pyrex) tubes with poly seal cone caps and filled to capacity with no air bubbles trapped. Samples should be transferred to the laboratory as quickly as possible. For high levels there is a possibility of loss of CO_2 and deposition of carbonate on the walls of the container and therefore containers should be filled to capacity. Bottles containing sludges should be filled only two thirds full.

Method

Dissolved inorganic carbon is measured colorimetrically by acidifying the sample and allowing the CO_2 produced to pass through a gas permeable membrane into a weakly buffered, alkaline, phenolphthalein solution. The decrease in absorbance of this colored solution is then measured. Chlorine and hydrogen sulphide interfere.

Time of Analysis

Approximately 150 dissolved inorganic carbon analyses can be performed per day.

CARBON - DISSOLVED ORGANIC

Carbon is the basic building block of all life. Inorganic and organic forms of carbon which enter the water from the atmosphere, surrounding lands and basins are interconverted during the natural life cycle.

The organic content of lakes and rivers is produced mainly by plants and animals which those water bodies support. Most of the organic carbon in water is composed of humic substances and partly degraded plant and animal materials, some of which are resistant to microbial degradation. The amount of organic carbon produced by photosynthetic processes will vary from season to season. Runoff from agricultural land, sewage, and industrial discharge from industries such as pulp and paper will add organic carbon to the water. The degradation of large amounts or organic matter causes depletion of the dissolved oxygen concentration which may be catastrophic for aerobic organisms in the receiving water body.

In natural waters, the organic carbon content will usually be less than 30 mg/L. This test may also measure some particulate carbon even though precautions are taken to minimize this.

Sampling

Samples should be collected in PET plastic containers. Samples should be transported to the laboratory as quickly as possible. Sludge samples should be filled only two thirds.

Method

The dissolved organic fraction is measured after it has been stripped of inorganic carbon and the sample has been oxidized by a U.V. digestor in an acid-persulphate medium. The dissolved organic carbon is then measured colorimetrically by allowing the CO_2 produced to pass through a gas permeable membrane into a weakly buffered, alkaline phenolphthalein solution. The decrease in absorbance of this colored solution is then measured.

Time of Analysis

Approximately 150 dissolved organic carbon analyses can be performed per day.

CHEMICAL OXYGEN DEMAND (COD)

The chemical oxygen demand is a measure of the oxygen required to oxidize susceptible organic and inorganic compounds under vigorous chemical oxidizing conditions. This test is especially useful for industrial wastes where the BOD_5 test is not applicable. Toxic materials in industrial wastes tend to inhibit bacterial activity producing a BOD_5 which generally underestimates the true organic composition of the sample. Most organic compounds are oxidized by the COD method. Benzene, toluene, pyridine, and similar aromatic ring compounds are incompletely oxidized. The COD may be used to estimate the effect of a particular waste on a receiving water or may be used to estimate the efficiency of a waste treatment process.

COD results do not necessarily relate directly to the BOD_5 value or to the oxygen consumption in the receiving water, but they do represent the ultimate carbonaceous oxygen demand that may eventually be exerted. If a number of comparative analyses show that a stable ratio exists between the COD and BOD_5 for a particular waste, then the COD results may be used to predict the approximate BOD_5 values.

Sampling

The COD is a perishable quantity since it may be influenced by bacterial activity. Immediate delivery to the laboratory is essential. Aqueous samples should be collected in PET plastic containers and refrigerated. Soil samples should be collected in pomade jars.

Method

Batches of samples are oxidized by oven digestion using dichromate in an acid medium. Following digestion, chromium III is detected using an automated continuous flow colorimetric system. For this technique the experimental conditions are adjusted to provide both a high and a low analytical range.

Time of Analysis

Approximately 80 COD analyses may be completed per day.

CHLORIDE

Chloride, in the form of the Cl^- ion, is a major anion in domestic wastes and in many natural water supplies. Urban runoff often contains high concentrations of chloride in winter due to the application of road salt.

Chloride poses no direct health hazard, but the water quality objective for domestic water is 250 mg/L of Cl to prevent a salty taste. This salty taste is variable and depends on the composition of the water. If chloride is present as sodium chloride, a detectable taste may be present at 250 mg/L, but if it is present as calcium or magnesium chloride there may be no noticeable taste at 1000 mg/L. A high chloride content may harm metallic pipes and structures as well as agricultural plants.

The colorimetric and titrimetric methods of chloride analysis actually measure chloride, bromide, iodide and cyanide. Therefore, caution must be exercised in interpreting the results. Generally, in natural waters, the concentrations of bromide, iodide and cyanide are not large enough to cause interference. In regions where watercourses are influenced by ocean tidewaters, or industrial discharge, these ions may provide appreciable interference.

Air samples are used to assess windborne chloride during the winter months.

Sampling

Water, sewage, industrial waste, precipitation and sludge samples should be collected in PET plastic containers. Collect soil samples in pomade jars, vegetation samples in plastic bags, and air samples using Hi-Vol sampler technique.

Method

Ground water and surface water samples are analyzed colorimetrically using mercuric thiocyanate and ferric ammonium sulphate. Bromide, iodide and cyanide interfere with the colorimetric test. Precipitations, dustfall and Hi-Vol samples are analysed by ion chromatography. Vegetation samples are analyzed by X-ray fluorescence, but it is elemental chlorine, not chloride, which is reported. (See matrix elements in vegetation).

Time of Analysis

Approximately 200 chloride analyses may be performed perday colorimetrically 30 by ion chromatography and 100 by x-ray.

CHLORINE - RESIDUAL

Chlorination of water supplies and polluted waters is performed to destroy or deactivate disease producing organisms. The chlorine may also react with ammonia, iron, manganese, sulphide and some organic substances resulting in improved water quality. Chlorination may produce adverse effects by intensifying the taste and odour characteristics of phenols and other organic compounds present in water.

Elemental chlorine is a gas which is slightly soluble in water and reacts readily with many inorganic substances and all plant and animal tissues. Liquified chlorine gas assumes three principal forms of free chlorine; hypochlorous acid, hypochlorite ion and aqueous molecular chlorine. Free chlorine reacts readily with ammonia and certain nitrogenous compounds to form combined available chlorines such as mono- and dichloramine. The presence and concentration of these combined forms depends on pH, temperature and the initial chlorine to nitrogen ratio. While treated waste waters may contain both free and combined forms, the free chlorine fraction is seldom found because it is generally added in a smaller amount than the demand of that water. Total residual chlorine (TRC) is the sum of the free available chlorine and the combined available chlorine.

In many water treatment plants, sodium hypochlorite is being used instead of chlorine gas because it is safer. Chlorine gas can be toxic, cause severe lung irritation and damage, irritate the eyes and can cause conjunctivitis. TRC levels of 0.014 to 0.029 mg/L can be toxic to caged rainbow trout. The Water Quality Objective states that levels should not exceed 0.002 mg/L in surface water because of toxicity to fish.

Sampling

Samples should be collected in PET plastic containers, filled to the top so that no air bubbles remain and kept refrigerated. Agitation or exposure to sunlight will accelerate the reduction of chlorine. Samples should be analyzed as soon as possible after collection and if possible, samples should be analyzed in the field. Consultation with laboratory staff is required prior to sampling as special preparations are necessary before the samples arrive, and authorization by laboratory staff is required before the test will be performed.

Method

Samples are analyzed by an amperometric titration. TRC, free available chlorine, monochloramine and dichloramine may all be measured by varying the amount, order and type of reagent. Nitrogen trichloride, organic chloramines, free halogens, copper, silver, surfactants, and some organics in highly colored waters may interfere with the various fractions. In addition, the forms of chlorine present in a sample may interfere with each other. Hence it is very important to notify the laboratory before sending samples.

Sewage and drinking water samples may also be analyzed for TRC using the Palin DPD comparator method.

Time of Analysis

Approximately 20 titrations can be performed per day. Time to perform one TRC by DPD comparator method is approximately 10 minutes.

CHROMIUM

Chromium appears in nature primarily as the mineral chromite or chrome iron ore. Chromium in rocks and soils is generally present as insoluble chromic oxide. During weathering, chromium behaves like iron and is retained in sands and clays. Thus little of the chromium actually goes into solution. Natural waters generally contain less than 0.001 mg/L. Chromium(III) and chromium(VI) are the two forms most common in waters. The hexavalent form is the most common since above pH 5, the trivalent ion precipitates out or gets oxidized to the hexavalent form.

Chromium can enter the waterways as a result of weathering or from industrial discharges from cement, ferrochromium, chromium steel, metal plating and leather and wood preservative industries. The burning of fossil fuels also contributes a large amount of chromium.

Chromium is present in small quantities in all soils and plants, but there is no evidence to indicate that it is required by plants for growth since chromic and chromate ions interfere with the uptake of essential elements. Soils with a high chromium content are generally infertile. Hexavalent chromium is the most toxic inorganic form of the element and has been demonstrated to exhibit carcinogenic properties. The maximum permissible concentration in domestic water supplies has been established at 0.05 mg/L of hexavalent chromium. Concentrations of chromium in unfiltered waters should not exceed 0.1 mg/L to protect aquatic life. The maximum allowable concentration of dusts and mists in air, measured as CrO_3 is 0.1 mg/m^3 per 8 hour exposure.

Sampling

Fill the sampling bottle completely and deliver it to the laboratory as quickly as possible. PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per litre of sample for total chromium analyses. Chromate ion is readily adsorbed on container surfaces and delays in shipment longer than 2 to 3 days may cause erroneous results.

Pomade jars are used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Glass bottles with teflon lined caps should be used for hexavalent chromium samples. No preservative should be used.

Method

Hexavalent chromium samples are analyzed colorimetrically with diphenylcarbazide with no sample pretreatment. High levels of chloride, vanadium, iron and mercury may interfere.

Total chromium samples are digested with the appropriate acid solution and analyzed by atomic absorption or Inductively Coupled Plasma (ICP) atomic emission (sewage, sludge and Hi-Vol samples).

Time of Analysis

Approximately 50 atomic absorption and 40 colorimetric analyses can be performed per day.

COBALT

Cobalt is found in nature in the minerals cobaltite, smaltite, and erythrite. Weathering of igneous and sedimentary rocks and soil leaching release cobalt to the water. Cobalt is also widely used in the manufacture of alloys and pigments used in glass staining. Recently it has found use as a binder in the tungsten carbide tool industry where it is commonly added to steel as a means of improving the cutting quality of certain tools. Cobalt is also used in the electroplating industry because of its appearance, hardness and resistance to oxidation. Levels in natural waters are generally lower than 0.0001 mg/L.

Cobalt is an essential element at trace levels for both animal and plant nutrition. It is known to be one of the main constituents of Vitamin B12 and its presence is believed to be instrumental in the natural synthesis of this vitamin. Cobalt deficiency in man and animals results in a type of anaemia which may be corrected by administering small dosages of cobalt chloride orally. Adverse effects due to cobalt are very slight even at high concentrations. Nausea and diarrhea are the common symptoms. No limits have been set on the maximum acceptable concentrations for cobalt in domestic water supplies. Tentative limits suggested by the Water Quality Criteria are 0.05 mg/L for continuous irrigation of all soils and 5 mg/L for use on fine textured soils. Cobalt is extremely toxic to Daphnia. The 48-hour LC-50 is 1.32 mg/L. Growth inhibition in carp has been observed above 5 mg/L.

Sampling

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per litre of sample. Pomade jars are used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Samples are digested with the appropriate acid solution and analyzed by atomic absorption.

Time of Analysis

Approximately 50 cobalt analyses may be performed per day.

COLOR - TRUE

Color in natural surface waters is primarily associated with the presence of humic acids derived from the decomposition of plant material such as plankton, aquatic macrophytes, terrestrial leaves and logs. Lakes containing these colored substances are commonly referred to as "acid-bog" or "brown-water" lakes. In ground waters, the color is more likely due to the presence of iron and manganese. In either case, the color is characteristically manifested as varying hues of yellowish-brown.

Water with colors of unusual hues, such as those of industrial origin, may differ greatly from the typical yellowish-brown color of natural water. Samples known to contain organic dyes, colored metallic compounds and other types of industrial effluents cannot be adequately assessed using this particular analytical technique.

When "true color" is reported, it represents only dissolved colored substances. "Apparent color" also includes the color contributed by suspended matter.

Waters colored naturally by humic substances are considered unacceptable for drinking purposes because of their appearance. If the color is due to the presence of iron and manganese, it can also be associated with taste and staining problems. Most naturally colored waters are harmless; hence the specified objective of 5 "true color" Units for domestic supplies is based on aesthetic considerations rather than on known health standards.

Sampling

Samples should be collected in PET plastic containers.

The color of a water is greatly dependent on pH, the extent of biological activity, and the precipitation of iron and manganese. To minimize changes during transit, sample containers should be filled to the neck, sealed tightly and refrigerated. Immediate delivery to the laboratory is essential.

Method

True colour is measured colorimetrically on the supernatant of a settled sample in a system calibrated with acidified chloroplatinate standards. The sample stream is measured using a broadband blue filter. Residual turbidity effects are suppressed by using a broadband red filter and increased path length in the reference stream.

Time of Analysis

Approximately 100 color analyses can be performed per day.

CONDUCTIVITY

Conductivity is defined as the reciprocal of a water's electrical resistance measured between two electrodes one square centimeter in area and one centimeter apart. In other words, conductivity is a measure of the ability of water to carry an electric current. The conductivity depends on the concentrations of the various ions in solution, the mobility of each of these ions and the temperature at which the measurement is made. All measurements are made at a standard temperature of 25°C.

Any aqueous system containing ionized substances will conduct an electric current. Most inorganic acids, bases and salts, such as hydrochloric acid, sodium bicarbonate and sodium chloride, are good conductors, while many organic compounds, such as glucose or benzene, undergo minimal dissociation and are poor conductors.

This test is so precise and accurate that it is often preferred to a dissolved solids test as an indicator of the dissolved solids content of a natural water sample. The conductivity may be correlated to the dissolved solids content (mg/L) by multiplying the conductivity by an appropriate factor. Generally, natural waters in Southern Ontario free of industrial wastes, have a factor of 0.65 ± .10. This factor may vary depending on the location.

Sampling

Aqueous samples should be collected in PET plastic containers. Soil samples should be collected in pomade jars.

Care must be taken to avoid contact with any material which can release or absorb ions. Samples should not be frozen as some carbonates are precipitated and are not easily redissolved.

Method

Measurements are made using a conductivity meter. A water bath is used to adjust the sample temperature to 25°C before the measurement is taken. Measurements can also be made on soil - water pastes, interstitial waters and leachates of soil, sediments and industrial wastes. Samples must not be diluted as there is a curvilinear relationship between dissolved solids and conductivity, particularly above 1,000 MS/cm. If samples are extracted with water, the conductivity relates only to those specific extraction volumes and conditions. Air bubbles and foreign material in the conductivity cell may lead to low results. Results are reported as microSiemens/cm (micromhos/cm).

Time of Analysis

Approximately 200 conductivity analyses can be performed per day on aqueous samples.

COPPER

Copper occurs naturally as the free native metal as well as in malachite, cuprite, chalcopyrite and various mixtures of sulphide ores. The presence of copper in natural waters is generally due to industrial activity, but in certain circumstances, copper salts may be introduced to a water distribution system or reservoir as a means of controlling objectionable algae slimes. Copper may also be introduced into the environment through contact with brass and copper plumbing, discharges from industries making textiles, paints and electrical products, and discharges from smelting operations and tailings ponds.

Total copper in water may occur as insoluble particulates, soluble complexes and soluble divalent ions. Copper concentrations in the Great Lakes are dependent on local inputs, but in open waters they seldom exceed 5 μg/L.

Copper is an essential element to the human body, acting as a catalyst in many biochemical reactions. It is generally agreed that copper is an active agent in the synthesis of hemoglobin and that a copper deficiency in animals causes a severe anemia similar to iron deficiency anemia. A daily intake of about 2 mg is required for normal body functions. Excessive amounts of copper are toxic, and cause abdominal pain, vomiting and convulsions. Prolonged exposure results in liver and kidney damage. Copper poisoning occurs more often in grazing livestock than in man, usually resulting from consumption of dry feeds and grasses with high copper concentrations. The free aquated copper ion is extremely toxic to fish. When bound by organic ligands, the metal is biologically less active. Copper imparts a disagreeable taste to water at concentrations well below those which would be hazardous to health; hence the drinking water standard of 1.0 mg/L is based on taste rather than on toxicity. Levels in unfiltered samples should not exceed 0.005 mg/L to protect aquatic life.

Sampling

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per litre of sample. Pomade jars are used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested in the appropriate acid medium and then analyzed. Sewage, sludge and Hi-Vol filters are analyzed by Inductively Coupled Plasma (ICP) atomic emission. All other matrices are analyzed by atomic absorption. Polarographic procedures may be used to differentiate "free" and "combined" copper.

Time of Analysis

Approximately 50 analyses can be performed per day by atomic absorption.

CYANIDE

Cyanides are a class of chemical compounds which contain a carbon- nitrogen constituent commonly referred to as the cyanide group. Cyanides vary in their degree of chemical activity depending on how readily they dissociate in water to form the cyanide ion. Common forms of cyanide are metal cyanide complexes, hydrocyanic acid and free cyanide ion from dissociated cyanide salts. Simple cyanides of the alkali metals readily form this ion while many metal cyanides do so at a much slower rate or not at all. The form of cyanide is also strongly dependent upon the pH. Most of the free cyanide exists as HCN at the pH values of natural waters.

The presence of cyanide in natural waters is the result of an industrial discharge from gas works, coke ovens, gas scrubbing in steel plants, electroplating industries, and precious metal refining operations. Waters containing this substance are extremely toxic to virtually all aquatic life forms and are unsafe for human consumption. Cyanide exerts its toxic effects by inhibiting the normal oxidation processes in body tissue thus causing asphyxiation and death. Chlorination processes can reduce cyanide levels in raw drinking water supplies. The maximum acceptable cyanide concentration for domestic water supplies in Ontario has been set at 0.01 mg/L of cyanide as HCN. Concentrations of free cyanide in an unfiltered sample should not exceed 0.005 mg/L to protect aquatic life.

Sampling

A sample suspected of containing cyanide should never by acidified, as this will release highly toxic hydrogen cyanide. Samples may be preserved with 1 mL of 50% NaOH per liter. Keep samples refrigerated and in darkness. PET plastic containers may be used for all types of samples, however, plastic bags are preferred for vegetation samples.

Method

Total Cyanide (CCNAUR)

Aqueous samples are distilled manually from a weakly acidic tartaric acid solution and "total cyanide" which is then determined colorimetrically by AutoAnalyzer using chloramine-T and a barbituric acid-isonicotinic acid reagent. Soluble cyanide in soils and sediments can be determined after extraction with distilled water.

Free Cyanide (CCNFUR)

Free or weak acid dissociable cyanide is determined in-line by a low temperature distillation (100°C) out of 10% acetic acid:.05% zinc acetate solution (pH4) and determined colorimetrically as above.

Time of Analysis

Approximately 75 samples for cyanide can be screened per day. Approximately 20 samples can be manually distilled per day.

DUSTFALL

Air pollutants in the form of dust can be a nuisance factor, since pollutants are readily perceived by the sensory organs and in some cases, the substances present in the dust may cause injury. By far the highest contributor to total dustfall is human activity. Wind occasionally contributes significant amounts.

Dust damages clothing and buildings, contaminates residential areas, damages agricultural crops and may cause eye injuries.

When assessing damages or injuries attributable to dust, an analysis of the elemental composition and a microscopic examination assist in targeting the possible toxic agents and their sources.

The ability of a dust particle to settle is mainly determined by its weight, size and surface area, wind and precipitation. The type of target also has an effect. Thus, dustfall data are affected by these external conditions. Results are used to express the atmospheric fallout of dust in specific areas.

Sampling

Dustfall is the settleable fraction of the in total particulate matter in air. It is collected in a polyethylene jar 15 cm diameter and 30 cm tall. The jar is exposed for approximately 30 days, 5 meters above ground level, usually on a suitable hydro pole.

Samples should be analyzed within one week of collection. To facilitate sample handling the Dustfall jar is lined with a polyethylene bag.

Method

The method closely follows the ASTM method for the collection and analysis of dustfall. The sample is filtered to obtain soluble and insoluble fractions. The fractions are dried and weighed and the calculations performed. Extraneous materials, such as insects, foliage, algae and fungi may interfere. Results are reported as $g/m^2/30$ days. All material that does not pass a 20 mesh screen is excluded.

Time of Analysis

Samples are run in batches of 36 and require 40 minutes per analysis.

FLUORIDATION RATE

Fluoride damage to vegetation is usually the result of airborne fluoride emissions which may cause chronic damage depending on the length and concentration of fumigation. The primary aluminum industry is the major source of atmospheric fluoride emissions. However, phosphate fertilizer production, elemental phosphorus plants and the primary iron, steel, and brick making industries are also significant contributors. Since the majority of fluoride emissions are in a gaseous state, such as hydrogen fluoride and silicon tetrafluoride, the fluoridation rate of the atmosphere is monitored.

Hydrogen fluoride gas severely irritates the eyes and respiratory system and may cause burns to the eyes. It may also cause irritation to the skin. The liquid causes severe, painful burns on contact with all body tissues.

The method for determining the fluoridation rate provides a relatively inexpensive surveillance method for monitoring the degree of pollution from gaseous fluorides in specified areas.

The criteria set for fluoridation rates are 40 mg F/100 cm^2/30 days for the summer growing season from April 15th to October 15th and 80 mg F/100 cm^2/30 days from October 16th to April 14th. Since the candle is exposed for 30 days, it is not possible to determine specific days of particularly high or low fluoride air pollution.

Sampling

Prior to and after exposure, the candle must be stored in the sealed tubular container provided.

Method

The fluoride is fixed during exposure of the candle by reaction with calcium oxide. The fluoride is extracted from the exposed filter paper in a buffer solution and is determined using a fluoride ion specific electrode in conjunction with a standard single junction, sleeve-type reference electrode.

Impacted fluoride particulates can lead to high results. Static monitors are subject to changes in wind speed and direction, temperature and humidity. Results are reported as mg F/100 cm^2/30 days.

Time of Analysis

One week is required to analyze each batch of candles. Approximately 27 candles can be prepared per day.

FLUORIDE

Fluoride occurs naturally in the ionic form in natural waters, and in soils and sediments primarily as the minerals fluorspar, fluoroapatite, fluorite and cryolite. Most surface waters in Ontario contain fluoride in concentrations less than 2 mg/L although higher levels may be found in localized areas containing fluoride bearing rocks. Ground water levels may be slightly higher than surface water levels.

While weathering processes supply a large portion of the fluoride in natural waters the presence of fluoride in drinking water is mainly due to the fluoridation of domestic water supplies. Fluoride concentrations near 1 mg/L are beneficial in retarding tooth decay. When fluoride is naturally present in drinking water, the concentration should not average more than 1.2 mg/L. Presence of fluoride in concentrations greater than 2.4 mg/L shall constitute grounds for rejection of the water supply. When fluoridation is practised, the permissible operating range is 1.0 mg/L to 1.4 mg/L. Fluoride concentrations between 2.4 and 10.0 mg/L can produce mottling of teeth and levels more than 50 mg/L may be toxic. Livestock drinking water should not exceed 2.0 mg/L and irrigation waters should contain less than 1.0 mg/L and 15 mg/L for use on acidic and neutral to alkaline soils respectively.

Fluoride is used in industry in a variety of manufacturing processes such as adhesives and metallurgical fluxes and may be contained in emissions from fertilizer, aluminum reduction, uranium processing and brick making operations.

Sampling

Aqueous samples should be collected in PET plastic containers and soil samples in pomade jars. Vegetation samples should be collected in perforated plastic bags and refrigerated.

Method

Water samples are distilled from sulphuric acid and analyzed colorimetrically on an AutoAnalyzer system by reaction with lanthanum nitrate and Alizarin Blue to form a ternary Alizarin Blue-lanthanide-fluoride complex. Vegetation samples are analyzed colorimetrically after extraction with 0.1 M perchloric acid. Vegetation, dustfall and Hi-Vol samples are analyzed with an ion specific electrode.

Time of Analysis

Approximately 100 aqueous samples can be analyzed for fluoride per day.

Gold is found most often in the metallic state as native gold but is also found with tellurides. It is widely distributed and is generally found with quartz or pyrite.

Gold is best known for its use as a monetary standard and in jewelry, decoration, dental work and plating. Gold is often used as an anticorrosive as a plating on other metals. Gold alloys are used in electronic apparatus. Clorauric acid is used in photography for toning the silver image. The Au-198 isotope is used for treating cancer and other diseases.

Sampling

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per liter of sample. Soil and sediment samples should be collected in pomade jars. Vegetation samples should be placed in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

The sample is digested with the appropriate acid mixture and analyzed by atomic absorption spectrophotometry.

Time of Analysis

Approximately 50 gold analyses can be performed per day.

HARDNESS

Water hardness is due mainly to the presence of calcium and magnesium salts in solution. However, aluminum, iron, manganese, strontium, zinc and hydrogen ions may contribute if present at high enough concentrations.

Hardness is reported as mg/L of calcium carbonate. Thus, the measured hardness is equal to the hardness which would be produced by the quoted concentration of calcium carbonate. The fraction of the total hardness accounted for by carbonate and bicarbonate salts in solution is called carbonate hardness and any excess is called noncarbonate hardness.

The hardness of waters varies widely throughout Ontario from Precambrian Shield levels of 20 - 75 mg/L, Lake Erie levels of about 130 mg/L to groundwaters which generally exceed 300 mg/L and may reach 2,000 mg/L.

Hard waters are objectionable because they form insoluble compounds with soap which greatly reduces the efficiency of the soap. At high temperatures they also form a lime scale in plumbing fixtures which can reduce the lifetimes of hot water pipes, boilers, heat exchangers, washing machines and any other appliances which use hot water.

Waters with levels above 500 mg/L are undesirable for both domestic and industrial uses.

Sampling

PET plastic containers must be filled completely so that no air bubbles remain once they are capped. This precaution will prevent the loss of dissolved carbon dioxide which is essential for keeping calcium carbonate in solution. Samples should not be refrigerated as this will cause precipitation of calcium carbonate.

Method

Hardness levels on domestic water and surface water samples are calculated from calcium and magnesium results obtained by atomic absorption.

Time of Analysis

About 200 samples per day can be analyzed for hardness concurrent with the associated calcium and magnesium analyses.

IRON (TOTAL)

Iron is the most abundant of the heavy metals in nature, but is generally found at low levels in most surface waters because of the relatively low solubility of ferric iron when the pH exceeds 7. Compounds of iron in water are in a state of dynamic chemical equilibrium caused by bacterial activity and chemical oxidation. Under reducing conditions, iron exists in the ferrous state. On exposure to the atmosphere or oxidants, ferrous iron is oxidized to the ferric state and may hydrolyze to form insoluble ferric oxides. Because the relative concentrations of these compounds are constantly changing, only a total iron determination provides relevant data. The most common forms of iron in water include ferrous ions, ferric ions, inorganic and organic chelates, complex colloidal organics, suspended oxides and hydroxides. Iron levels in surface waters are generally less than 0.5 mg/L but they often exceed this in groundwaters. Iron is added to the environment by weathering of iron bearing minerals such as pyrite, hematite and magnetite, mineral processing and acid mine drainage.

The role of iron is important in eutrophication studies as it affects retention and release mechanisms of phosphorus components in lake and river sediments. This ability of iron to bind up phosphorus is also important in nutrient removal programs at water pollution control plants where ferric chloride is sometimes used as a phosphate precipitant.

Iron is an important component of hemoglobin and an essential element for all life forms. It is non-toxic at high levels but is objectionable in domestic supplies because of the color and bitter taste it imparts. It is also known to cause reddish-brown stains on plumbing fixtures and laundry. The water quality objective for iron in domestic water supplies is 0.3 mg/L (filterable). Levels in an unfiltered sample should not exceed 0.3 mg/L to protect aquatic life.

Sampling

Only total iron analyses are performed routinely in water samples. If filterable iron is requested, samplers must request a total iron analysis on a field filtered sample. PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per liter of sample. Pomade jars are used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Samples are digested with an appropriate acid mixture and analyzed by Inductively Coupled Plasma (ICP) atomic emission or by atomic absorption spectrophotometry.

Time of Analysis

Approximately 50 iron analyses can be performed per day.

LEAD

Lead is found in nature primarily as the mineral galena (lead sulphide) but also as cerussite, anglesite, crocoite, wulfenite, pyromorphite, matlockite and vanadinate. Since the inorganic salts of lead are relatively insoluble, the element most commonly enters a water supply via industrial, mine and smelter discharges or through dissolution of old corroded lead plumbing. The latter problem is much more common in areas where the water is "soft"; that is, it has a low hardness or mineral content. Other sources of lead include the burning of leaded fuels and the production of lead batteries, explosives and paints. Levels in surface waters generally are below 0.04 mg/L.

Lead is an extremely toxic element which tends to exchange with calcium and accumulate in bone marrow. Lead toxicity may not appear until many years after the last exposure. Organic compounds of lead are many times more toxic than inorganic forms. The most common example is tetraethyl lead which is used as an "antiknock" additive in motor fuels. Organic lead exerts its toxic effect on the nervous system within a very short time causing mental confusion, delirium, nausea, hallucinations, insomnia, and convulsions. The maximum acceptable concentration of lead in domestic water is 0.05 mg/L. The toxicity of lead to aquatic life is highly dependent on the alkalinity. The toxicity generally decreases as the alkalinity increases. The total lead concentrations should not exceed the values below.

Alkalinity mg/L as $CaCO_3$	Maximum Lead Concentration µg/L
< 20	5
20-40	10
40-80	20
> 80	25

Sampling

When levels below the normal range of application are required, a notation must be included on the sample submission form so that the sample will be preconcentrated prior to analysis. 500 mls is required exclusively for the preconcentration process.

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per liter of sample. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested with the appropriate acid mixture and analyzed. Sewage and sludge samples are analyzed by Inductively Coupled Plasma (ICP) atomic emission. Hi-Vol samples are analyzed by X-ray fluorescence. All other samples are analyzed by atomic absorption. A polarographic method can also be used for low concentrations in some aqueous matrices.

Time of Analysis

Approximately 50 lead analyses can be performed per day by Inductively Coupled Plasma (ICP).

LITHIUM

The element lithium is comparatively rare. It is concentrated in complex silicate minerals such as petalite and spodumene. Lithium is easily weathered and because lithium compounds are very soluble, they tend to stay in solution. It is rarely found in surface waters and water supplies, but it has been detected in hot springs and highly mineralized brines where the concentration is usually less than 10 mg/L. The major source of lithium in natural waters is industrial discharge.

Lithium is used in alloys, organic syntheses and special glasses. Lithium chloride and lithium stearate are used in de-humidifiers. Lithium hydride is very corrosive and irritating.

Lithium becomes toxic to animals and humans only if taken in very large amounts. When in sufficient quantity, it has the capacity for replacing sodium in body cells and thus upsetting intra-cellular metabolism. Cause of death is generally due to kidney failure although secondary effects on the nervous system are also observed. Small doses of lithium carbonate have been effectively used as an anti-depressant for treatment of psychiatric patients. No guidelines have yet to be set for drinking waters, but irrigation water must contain less than 2.5 mg/L.

Sampling

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per litre of sample. Soil and sediment samples should be collected in pomade jars. Vegetation should be placed in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested with the appropriate acid mixture and analyzed by atomic absorption spectrophotometry.

Time of Analysis

Approximately 50 lithium analyses can be performed per day.

MAGNESIUM

Magnesium ranks eighth in the natural order of elemental abundance and is commonly found in the minerals magnesite, dolomite, olivine, serpentine, talc and asbestos. Its industrial applications include the manufacture of metal alloys, photographic flash bulbs, flares, cement, tanning solutions and explosives. It is also used in milk of magnesia and epsom salts.

Magnesium is present in all natural waters and is generally associated with the presence of calcium. In natural fresh water systems the calcium concentration is approximately three to five times that of magnesium. If the reverse is found, an artificial source of magnesium is probable. Natural processes generally add more magnesium to the environment than do industrial processes.

Magnesium is an essential element for the life functions of all plants and animals. It is closely associated with the metabolic activity of phosphorus where it is believed to act as an activator of phosphate enzymes. Ingestion of magnesium produces no adverse effects at levels normally encountered in domestic water supplies, but concentrations in excess of 125 mg/L exert a cathartic action on the gastro-intestinal tract.

Magnesium is a major contributor to water "hardness"; therefore limitations on the amount of magnesium in water supplies are of concern especially to industry (see Hardness). Magnesium may also impart a disagreeable taste.

Sampling

Routine surface, domestic water and precipitation samples should be collected in PET plastic containers.

Non-routine industrial waste samples, or any other samples where high levels of heavy metals are suspected, should be sampled in PET plastic containers and preserved with 20 drops of nitric acid per liter. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Drinking water, surface water, and precipitation samples are analyzed directly by atomic absorption. Other samples are digested with an appropriate acid mixture depending on the sample matrix and the laboratory section doing the work. Sewage samples are analyzed by Inductively Coupled Plasma (ICP) atomic emission. All other matrices are analyzed by atomic absorption.

Time of Analysis

Approximately 200 magnesium analyses can be performed per day by atomic absorption and 50 per day by Inductively Coupled Plasma (ICP).

MANGANESE

Manganese is a common element in nature and found in numerous minerals as an oxide, sulphide, carbonate and a silicate. It is found at very low levels in natural waters, generally in the divalent (soluble) or quadrivalent (insoluble) state, but in the absence of dissolved oxygen, the divalent state predominates. The permanganate anion, containing manganese in the heptavalent state, is not found in water unless it has been added artificially. This test does not distinguish between the various oxidation states of manganese. High levels of manganese are usually the result of industrial discharges. Common industrial applications include the manufacture of metal alloys, dry cell batteries, paints, varnishes, inks, dyes, glass, ceramics, matches, fireworks, and fertilizer. Iron and steel plants and acid mine drainage account for a large portion of the manganese found in the environment.

Manganese in trace quantities is essential for the proper nutrition of both animals and plants and is believed to be important in the metabolic oxidation-reduction processes of living cells. Manganese deficiency in animals is characterized by lack of growth, bowing and abnormal fragility of the bones, and degeneration of the reproductive organs. Manganese and its salts, ingested in very large quantities, are potent nerve toxins.

Although manganese is non-toxic at the levels commonly encountered in water supplies, it causes unpleasant tastes, stains laundry and plumbing fixtures, and encourages the growth of objectionable micro-organisms at treatment plants. The maximum acceptable concentration of manganese for domestic water supplies in Ontario is 0.05 mg/L.

Sampling

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per liter of sample. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Samples are digested with an appropriate acid mixture and analyzed by Inductively Coupled Plasma (ICP) atomic emission or by atomic absorption spectrophotometry.

Time of Analysis

Approximately 150 manganese analyses can be performed per day by the colorimetric method and 50 by atomic absorption.

MATRIX ELEMENTS IN VEGETATION BY X-RAY FLUORESCENCE

Vegetation is composed mainly of carbon, hydrogen, oxygen, nitrogen and eight "matrix elements". These matrix elements are considered to be sodium, potassium, calcium, magnesium, silicon, phosphorus, sulphur and chlorine as these elements are typically found in concentrations in excess of 1000 ppm. A comparison of matrix element concentrations in damaged vegetation with those considered normal for a particular species provides a relatively rapid method of assessing possible pollution damage to vegetation.

Potassium, calcium, silicon, phosphorus, sulphur and chlorine are measured by this procedure, only potassium, sulphur and chlorine are routinely reported.

Several other tests are performed on vegetation samples. For additional material, the reader is referred to the appropriate individual test outlines.

Sampling

At least 2 grams of dried, ground vegetation are required. Samples should be collected in perforated plastic bags, refrigerated and transported to the laboratory as quickly as possible.

Method

Samples are pelletized and analyzed by measuring the fluorescent intensity for each of the six elements for 8 seconds. A linear calibration is used to convert intensity readings to concentrations. The concentrations are corrected for inter-element effects.

Time of Analysis

Approximately 100 samples can be weighed and pelletized per day. Approximately 140 samples can be analyzed for all six elements per day.

MERCURY

Mercury is a relatively rare element often found as the mineral cinnabar (mercuric sulphide). Mercury is seldom present in natural waters at levels exceeding 50 parts per billion. The solubility of metallic mercury in water is low and the solubility of ionic mercury compounds depends upon the actual conditions of the solubilizing water (i.e. acidity, presence of complexing anions, other organo-metallic complexes). As a result, the mercury content of surface water depends upon the accessibility of mercury, time of contact and conditions of the solubilizing media.

Mercury has many industrial applications which include (in order of consumption) electrical apparatus, chloralkali industry, paint, industrial control instruments, agriculture, dental applications, general laboratory use, and and pulp and paper production.

The greatest environmental hazard associated with mercury is its tendency to become concentrated in living organisms. Any form of mercury entering an aquatic system could be converted via bacterial activity to methyl mercury derivatives. These substances can then become concentrated along a food chain until they reach toxic levels and begin to endanger higher forms of life. In man, mercury exerts its toxic effects on the nervous system. Common symptoms include muscular tremors, speech impairment, lack of co-ordination and emotional disturbances. The maximum acceptable concentration for mercury in drinking water is 1.0 μg/L.

Sampling

Aqueous samples should be collected in special milk dilution (#8C) glass bottles. Preservation is accomplished by adding 1 mL of concentrated nitric acid (preferably Baker analyzed) per bottle and 0.5 mL of potassium dichromate solution (saturated) to maintain a slight yellow color. Sludge samples should be collected in glass jars (two thirds full). Soil and dried vegetation samples should be collected in pomade jars. Fish samples should be placed in plastic bags and frozen. Immediate delivery to the laboratory is essential.

Concentrations of total mercury in *filtered* water should not exceed 0.2 μg/L nor should the concentrations of total mercury in whole fish exceed 0.5 μg/g (See Table 2).

Method

Samples ar e and analyzed by cold vapour flameless atomic absorption.

Time of Analysis

Approximately 30 mercury analyses can be performed per day.

MICROBIOLOGICAL INDICATORS OF FECAL POLLUTION

Escherichia coli (E. coli)

E. coli is the predominant, facultative bacterial species in the large bowel and is thus the coliform most directly related to fecal pollution. E. coli is occasionally pathogenic to man (e.g. urinary tract infections) but its primary importance in water bacteriology is as an indicator. Epidemiological studies conducted by the USEPA have shown that the concentration of E. coli in fresh recreational water is correlated with the onset of gastrointestinal disease in swimmers.

Methods developed historically for the detection of fecal pollution have not been sufficiently specific for E. coli. However, recently developed media designed specifically for the detection of E. coli have been tested in this laboratory and proven successful. E. coli is of value as a specific indicator of fecal contamination and of risk to recreational water users.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

E. coli levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within 24 hours, but not exceeding 48 hours.

Method

Analysis for E. coli requires filtration of an appropriate volume or dilution of the sample and incubation on mTEC Agar. The plates are incubated to allow a gradual increase to 44.5 ± 0.5°C over approximately 4 hours. Total incubation time is 23 ± 1 hours.

When using mTEC the filter is then transferred to a filter pad saturated with urease reagent and allowed to stand for 15 minutes. Urease positive colonies will turn magenta. Using a stereoscopic microscope (10X), all colonies that remain yellow, yellow-green or yellow-brown, are counted as E. coli. Modified membrane filter (MF) or most probable number (MPN) methods are available for the analysis of samples containing significantly high concentrations of FC (non E. coli) relative to E. coli and/or concentrations of Urease negative Klebsiella sp. **The modified tests require prior consultation and are not available for routine work.**

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, and the incubation period. E. coli results will be available 2 days after sample submission.

MICROBIOLOGICAL INDICATORS OF FECAL POLLUTION

Enterococci (ENT) Procedure

The ENT group of bacteria is a sub-group of the FS consisting of the variants of S. faecalis, S. faecium and S. durans. The primary source for these bacteria is feces. Studies by the US E.P.A. have shown a correlation between increasing concentrations of ENT in surface water and increasing incidence of gastrointestinal disease in swimmers.

The presence of Enterococci indicates the impact of fecal pollution on recreational waters and can be used as an indicator of risk to users. It is, however, subordinate in use to E. coli which is a more specific indicator of Fecal pollution.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or rubber syringes. Samples should be packed in ice but should not be frozen.

ENT levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within 24 hours, but not exceeding 48 hours. The necessary frequency of sampling is outlined in the MOE Water Management booklet (1984).

Method

Analysis for ENT is by membrane filtration (HAMES). The membrane is placed on m-E IG Agar and incubated at 41.5 ± 0.5°C for 48 ± 3 hours. Using a stereoscopic microscope (10X), all colonies that are grey-blue to maroon with a blue halo are counted as Enterococci.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, the number of parameters requested and the incubation periods for each parameter. ENT results are usually available within 72 hours of submission.

MICROBIOLOGICAL INDICATORS OF FECAL POLLUTION

Fecal Coliforms (FC) Procedure

The FC group of bacteria is basically a subgroup of the Total coliforms and is comprised of aerobic and facultative anaerobic, Gram-negative, nonsporeforming, rod-shaped bacteria which ferment lactose at 44.5°C. The FC parameter is a more specific test for bacteria associated with human and animal fecal material. This test has proved useful as an indicator of relatively recent fecal pollution inputs, however, it is by no means completely selective for Escherichia coli, the coliform most directly related to fecal pollution. Bacteria related to other forms of input such as Klebsiella may be detected with this test. The procedure is of value in detecting the presence of fecal pollution providing E. coli has been established as the major fecal coliform present.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or rubber syringes. Samples should be packed in ice but should not be frozen.

FC levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within 24 hours, but not exceeding 48 hours. The necessary frequency of sampling is outlined in the MOE Water Management booklet (1984).

Method

Analyses for fecal coliforms are by membrane filtration. The membrane is placed on a petri dish containing mTEC agar and incubated to allow a gradual increase to 44.5 ± 0.5°C over a four hour period. The total incubation time is 23 ± 1 hours.

After incubation the plates are kept at room temperature for 15 minutes and then, the number of fecal coliform colonies are determined with a stereoscopic microscope at 10X magnification. All yellow, yellow- brown and yellow-green colonies are counted.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, the number of parameters requested per sample and the incubation periods for each parameter. FC results are generally available within 48 hours of submission.

MICROBIOLOGICAL INDICATORS OF FECAL POLLUTION

Fecal Streptococci (FS) Procedure

The FS group of bacteria is comprised of aerobic, spherical to ovoid Gram positive cells occurring singly, in pairs or in short or long chains. When used as indicators of fecal contamination, the following species and varieties are implied: *Streptococcus faecalis*, *S. faecalis* var. liquefaciens, *S. faecalis* var. zymogenes, *S. faecium*, *S. faecium* var. casseliflavus, *S. durans*, *S. bovis*, *S. equinus*, and *S. avium*.

The fecal streptococci procedure is used as an indicator of fecal pollution but because of the presence of environmental strains, particularly during storm events, it should be used primarily as a backup to FC/*E. coli* results.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or rubber syringes. Samples should be packed in ice but should not be frozen.

FS levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within 24 hours, but not exceeding 48 hours. The necessary frequency of sampling is outlined in the MOE Water Management booklet (1984).

Method

Analysis for FS is by membrane filtration (HAMES). The membrane is placed on m-Enterococcus Agar (Difco) and incubated at 35.0 ± 0.5°C for 48 ± 3 hours. Using a stereoscopic microscope (10X), all colonies that are red, maroon or pink are counted as fecal streptococci.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, the number of parameters requested and the incubation periods for each parameter. FS results are usually available within 72 hours of submission.

Presence-Absence (P-A) Procedure

The P-A procedure has been developed as an alternative to the membrane filtration procedure and is a sensitive means of detecting pollution indicator bacteria in drinking water samples. Essentially, the P-A test is a modification of the most probable number (MPN) procedure, which uses a dilution series of broth tubes to detect and enumerate microorganisms. The P-A test employs only one analysis bottle per sample. The test allows for an inoculum of up to 100 mL and will qualitatively determine a wide variety of pollution indicator bacteria. They include total coliforms, fecal coliforms, Escherichia coli fecal streptococci, Pseudomonas aeruginosa, Staphylococcus aureus, Aeromonas sp. and Clostridium perfringens.

Sampling

Samples should be collected in pre-sterilized bottles with red labels (thiosulphate added). Samples should be packed in styrofoam containers, which include a ice bottles during the summer months.

Bacterial levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within 24 hours, but not exceeding 48 hours if feasible. Sample analysis will not be performed on samples older than 3 days. The necessary frequency of sampling is outlined in the MOE Drinking Water Objectives (1983) booklet.

Method

The P-A test involves the inoculation of 100 mL of water into a bottle of Lactose Lauryl Sulphate broth (HAMES). The bottle is incubated at 35 ± 0.5°C for up to four days. If growth occurs with the production of acid and/or gas, then other tests are used to determine the types of bacteria present.

Time of Analysis

The time required for completion of test procedures associated with the P-A test is up to one week depending on the level of pollution in the sample. A sample containing a variety of indicator bacteria will require several confirmation tests before results can be reported. Drinking water samples which show the presence of fecal bacteria within 24 hours will have this information phoned to the municipality as soon as confirmed results are available.

MICROBIOLOGICAL INDICATORS OF FECAL POLLUTION

Pseudomonas aeruginosa (P. aeruginosa) Procedure

P. aeruginosa is a motile, Gram-negative rod which does not ferment lactose and is oxidase positive.

Isolates of *P. aeruginosa* are found in feces and their presence in water is generally the direct cause of recent fecal waste inputs from a nearby point source. Their presence is thus a good indication of a potential health hazard.

P. aeruginosa is also an opportunistic pathogen and has been identified as the causative agent of a number of infections that may be transmitted through a contaminated water body to a susceptible host. Of chief concern to bathers is the association between *P. aeruginosa* and the disease known as otitis externa "swimmer's ear".

The presence of P. aeruginosa is a good indication of point source inputs of fecal contamination eg. combined sewers, faulty septic systems and of direct health risk to swimmers.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

P. aeruginosa levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within 24 hours, but not exceeding 48 hours.

Method

An appropriate volume or dilution of the sample is filtered through a 0.45 µm membrane filter and incubated on a modified mPA Agar at 41.5 ± 0.5°C for 48 ± 2 hours.

Using a stereoscopic microscope (10X), all tan to dark-brown or black colonies are counted as *Pseudomonas aeruginosa*.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, the number of parameters requested and the incubation periods for each parameter. *P. aeruginosa* results are usually available 72 hours after submission.

MICROBIOLOGICAL INDICATORS OF FECAL POLLUTION

Salmonella - Non-routine Procedure

Salmonella is one of the most important and common waterborne pathogenic organisms. The members of this genus are widely distributed in the environment and are probably responsible for most of the recognized waterborne disease outbreaks. Salmonella can be frequently detected in sewage, stormwater and polluted rivers and lakes. Fecal contamination from animals, particularly domestic, and insufficiently treated human wastes are the major source of these organisms.

Since Salmonella serotypes are all potentially pathogenic, their frequent isolation is taken as proof of a public health hazard.

The methods required are complex and slow, therefore their use is recommended only for specific studies and only with consultation and careful planning.

Salmonella detected in environmental samples indicates the presence of fecal contamination and a significant health hazard.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added). Samples should be packed in ice but should not be frozen.

Sampling can also be carried out with Moore Swabs (gauze pad) placed at appropriate locations in sampling areas.

Salmonella levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 24 hours.

Method

There are several procedures for the isolation and identification of Salmonella from waters and wastewaters. The method chosen will depend on the survey conducted. All the procedures include sample concentration (Moore swab or membrane filtration), enrichment in selective broths, plating on selective agar media and identification by a variety of biochemical and serological tests.

Time of Analysis

The number of samples analyzed will depend on the volume and type of sample and, more specifically, on the methods necessary for analysis. In general, a five-day period is required for Salmonella analyses.

Consultation with Microbiology staff prior to the use of this parameter is absolutely essential.

ROUTINE

Total Coliforms (TC) Procedure

The TC group is comprised of all of the aerobic and facultative anaerobic, Gram-negative, non-sporeforming, rod-shaped bacteria which ferment lactose with gas formation within 48 hours at 35°C or all organisms which produce a colony with a gold-green metallic sheen within 24 hours of incubation using the membrane filter (MF) technique. All coliforms are oxidase negative. This definition includes, in addition to the intestinal forms of the Escherichia coli group, closely related bacteria of the genera Klebsiella, Citrobacter and Enterobacter. Enterobacter - Citrobacter groups are common in soil but are also recovered from feces in small numbers. Therefore, the presence of total coliforms in water is not a specific indicator of sewage or fecal pollution but may also indicate runoff from soil. Its prime value is in the evaluation of water for human consumption eg. municipal distribution systems.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

TC levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within 24 hours, but not exceeding 48 hours. The necessary frequency of sampling is outlined in the MOE Water Management booklet (1984).

Method

Analysis for TC is by membrane filtration according to the MOE Handbook of Analytical Methods for Environmental Samples (HAMES). A given volume of the sample or dilution is filtered using a 0.45 µm gridded, white membrane filter (e.g. Gelman GN-6). The membrane is placed on m-Endo Agar LES (Difco) and incubated for 22 ± 2 hours at 35 ± 0.5°C. After incubation, the number of total coliforms and background colonies are counted using a stereoscopic microscope at a 10X magnification.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, the number of parameters requested per sample and the incubation periods for each parameter. TC results are generally available within 48 hours of submission.

Fungi (Yeasts and Molds)

Fungi are ubiquitously distributed, chlorophyll-free, heterotrophic microorganisms which are present wherever living or non-living organic matter occurs. Both saprophytic and pathogenic fungi have been found in sewage and sludge, domestic and industrial effluent and marine and freshwater environments. The densities and distribution of species of fungi in such habitats can be used as an index of the degree of organic enrichment. In addition, the presence of certain pathogenic fungi (e.g. Candida albicans) in recreational waters is useful in assessing water quality and in evaluating public health hazards.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

Fungal levels, yeasts in particular, will undergo change even upon refrigeration, therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 24 hours.

Method

No standard techniques have been developed for quantitative enumeration of fungi from an aquatic environment. In general, selective agar media, the membrane filtration (MF) technique and the spread plate technique are used. Fungal densities are expressed as colony-forming units (CFU) per 100 mL.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required and the incubation periods. In general, the analysis requires a five-day period.

Prior consultation is required before this parameter can be requested.

MICROBIOLOGICAL INDICATORS OF ORGANIC ENRICHMENT

Heterotrophic Bacteria - 20°C (HB-20) - Surface water

The HB-20 count is not a test for a specific microorganism. It is designed to enumerate as large a number as possible of those bacteria in water that require some organic carbon for their growth. The concentration of heterotrophic bacteria in water is affected by the levels of organic and inorganic nutrients present. Densities of HB-20 in lakes of high trophic status are higher than HB-20 levels in oligotrophic lakes.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

HB-20 levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 24 hours.

Method

Analysis is by the spot plate or spread plate method. Two volumes of 0.1 mL of the sample and/or sample dilution are placed on pre-dried CPS agar plates. The spread plate method requires one volume of 0.1 to 1.0 mL spread over the surface of the pre-dried agar plate. Plates are incubated at 20 ± 0.5°C for 7 days. Using a stereoscope microscope (10X), a total colony count is obtained.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, and the incubation period. HB-20 results by this method are available 8 days after sample submission.

Heterotrophic Bacteria - 35°C (HB35 MF)

The HB35 MF parameter can be used for the analysis of treated waters (i.e. drinking waters, water in distribution systems, bottled water) and untreated well water. In these waters the HB35 MF parameter is used to measure treatment efficiencies and as an indicator of general bacterial water quality.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added). Samples should be packed in ice but should not be frozen. HB35 MF levels will undergo change even upon refrigeration; therefore immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not more than 24 hours.

Method

Analysis is performed by filtering 1 mL through a membrane filter (MF) which is placed on heterotrophic plate count (m-HPC) agar. Standard Methods (16th Edition) recommends an incubation period of 48 ± 3 hours (bottled water for 72 ± 4 hours) at 35 ± 0.5°C. A total colony count which exceeds 500 organisms per mL is considered to represent poor water quality.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, and the overall laboratory workload. HB35 MF results are available 3 to 4 days after the sample is submitted.

MICROBIOLOGICAL INDICATORS OF

INDUSTRIAL/AGRICULTURAL POLLUTION

Klebsiella pneumoniae (K. pneumoniae) - Presumptive

K. pneumoniae is frequently found in the respiratory, intestinal and urogenital tracts of humans, but makes up only a small proportion of fecal coliforms in feces. These organisms may also be isolated from a variety of animals and materials and are well distributed in the environment. It is associated primarily with water high in organic matter. Thus, K. pneumoniae can be used to determine the extent of influence of certain industrial wastes such as pulp and paper mill wastes. A further concern, which still has to be proven epidemiologically, is that K. pneumoniae which is an opportunistic pathogen, could infect humans and cause infections or even a highly fatal type of pneumonia which accounts for 3% of bacterial pneumonias.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

K. pneumoniae levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within 24 hours, but not exceeding 48 hours.

Method

Presumptive analysis for Klebsiella pneumoniae is by membrane filtration (MF) of an appropriate volume or dilution of a sample with incubation on modified mK Agar at 35 ± 0.5°C for 48 ± 3 hours. Using a stereoscopic microscope (10X), all non-red colonies larger than 2 mm in diameter are counted as Klebsiella.

To confirm the presence of K. pneumoniae isolates must be obtained from the MF colonies and tested for gas production in EC broth at 44.5°C.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, and the incubation period. Presumptive K. pneumoniae results will be available 3 days after sample submission. Prior consultation is required before a request for a confirmed analysis can be considered.

MICROBIOLOGICAL INDICATORS OF INDUSTRIAL/AGRICULTURAL POLLUTION

Nitrogen Cycle Bacteria

Several groups of bacteria contribute extensively to the transformations occuring within the nitrogen cycle, both in aquatic and terrestrial environments. These groups include: a) the ammonifiers, comprised mainly of heterotrophic bacteria, which release ammonia from decaying proteinaceous material; b) the nitrifying bacteria, comprised of the genera Nitrosomonas and Nitrobacter, which, by an autotrophic, aerobic process oxidize ammonia to nitrite and nitrite to nitrate; c) the denitrifying bacteria, a diverse bacterial group, which through mainly anaerobic processes, remove nitrate from the environment either by reduction to nitrite or ammonia, or by transformation of nitrate to nitrogen gas; and d) the nitrogen-fixing bacteria, i.e. Azotobacter, which transform nitrogen gas into ammonia nitrogen.

This is a natural nutrient cycle and is sensitive to alterations in the environment such as increased acidity, toxic materials etc. The enumeration, of nitrogen cycle bacteria complemented by activity studies, reflects the potential of an aqueous environment to mineralize proteinaceous nitrogen (ammonifiers, nitrifiers and denitrifiers) or the ability to supplement nitrogen requirements through nitrogen fixation. The detection of these bacteria in water and wastewaters are not necessarily used as an indication of deteriorating water quality. However, elevated levels of nitrifying bacteria can indicate excessive loading with ammonia-nitrogen and an associated increased oxygen demand.

Methods presently available in the MOE are only for the enumeration of nitrifying and denitrifying bacteria. These tests are of most use in establishing zones of influence of industrial/agricultural inputs where fecal pollution is not of concern.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

Nitrogen cycle bacterial levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, but not exceeding 48 hours.

Method

Nitrogen cycle bacteria are determined by Most Probable Number (MPN) analyses in selective media broths. Incubation for the nitrifiers is at 28 ± 0.5°C for 28 days under aerobic conditions. Denitrifiers are incubated anaerobically at 28 ± 0.5°C from 7 to 21 days.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, the number of parameters requested and the incubation periods for each parameter. Nitrosomonas and Nitrobacter results are available 29 days after sample submission. Denitrifier results are available in 8 to 22 days depending on the incubation time. **Prior consultation with Microbiology staff is necessary before requesting these parameters.**

MICROBIOLOGICAL INDICATORS OF

INDUSTRIAL/AGRICULTURAL POLLUTION

Phenol Degrading Bacteria

The phenol degrading bacteria are a small heterogeneous group of bacteria that can degrade phenols if the concentration does not exceed 2 g/L. These bacteria are not monitored in order to detect a microbial hazard but to help determine the possible consequences of pollution by phenol and related compounds.

Pollution of surface waters with phenolic substances occurs as a result of their extensive industrial use. The presence of phenolics in surface waters can pose a serious pollution threat due to the toxic nature of these compounds. The potential for serious damage to the environment is increased if the area affected does not contain significant concentrations of bacteria capable of degrading these chemicals.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

The concentrations of phenol degrading bacteria will undergo change even upon refrigeration; therefore immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, but not exceeding 24 hours.

Method

Phenol degrading bacteria levels are determined by Most Probable Number (MPN) analyses in a broth containing phenol as the sole carbon source. Incubation is at 35 ± 0.5°C for 72 hours under aerobic conditions.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, the number of parameters requested and the incubation periods for each parameter. Phenol degrading bacteria results are available four days after sample submission. **Prior consultation before submitting samples is necessary.**

MICROBIOLOGICAL INDICATORS OF

INDUSTRIAL/AGRICULTURAL POLLUTION

Sulphate-Reducing Bacteria

The sulphate-reducing bacteria e.g. (Desulfovibrio, Desulfotomaculum) are a group of organisms which obtain energy from the reduction of sulphate to sulphide and grow in anaerobic environments which contain organics and a supply of sulphate. These organisms contribute to the deterioration of water quality by their production of H_2S and their capability of maintaining anaerobic and reducing environments.

The organisms have an economic importance in their contribution to the corrosion of iron pipes and steel containers, spoilage of stored petroleum products and the blackening of wood pulp.

Sulphate-reducers react to changes in concentration of reducible sulphates in the environment and are useful in detecting zones of influence of industrial wastes such as pulp and paper mill effluents.

Sampling

Samples should be collected in pre-sterilized glass bottles or rubber syringes. Bottles with thiosulphate added may be used **but prior consultation with laboratory staff is requested.**

Samples should be packed in ice but should not be frozen.

Sample containers should be as full as possible and tightly sealed to prevent oxidation. Do not leave an air space.

Sulphate reducer levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 48 hours.

Method

Sulphate reducers are determined by a Most Probable Number (MPN) technique. Using tubes of API broth with tryptone, a 3 x 3 series is set up using three appropriate volumes (weights) and/or dilutions of the sample (e.g. 3 tubes each of 10 mL, 1 mL and .1 mL). The tubes are layered with paraffin oil, to provide anaerobic conditions and incubated for 21 days at $28 \pm 0.5^{\circ}C$. A black precipitate is taken as a positive reaction.

Time of Analysis

The number of analyses performed per day will depend on the type of sample and the number of dilution steps required. Sulphate reducer results are available 22 days after sample submission. **Prior consultation with Microbiology staff is required before this parameter can be requested.**

MICROBIOLOGICAL INDICATORS OF

INDUSTRIAL/AGRICULTURAL POLLUTION

Sulphur-Oxidizing Bacteria

The sulphur-oxidizing bacteria include heterotrophic bacteria such as Pseudomonas sp, and members of the bacterial genus Thiobacillus which are a group of aerobic, autotrophic organisms (T. thioparus, T. thiooxidans and T. ferrooxidans) which derive energy from the oxidation of elemental sulphur, sulphide or thiosulphate. These organisms are able to multiply and maintain acidic environments. They contribute to the deterioration of water quality by lowering the water pH when the water body is enriched with reduced sulphur compounds and are responsible for the formation of acidic conditions in leachates of mine-tailings containing sulphide-based ores. Their activity also contributes to the corrosion of concrete in sewers and structures immersed in water. Their major use is in conjunction with sulphate-reducers to detect changes in the sulphur cycle brought on by chemical inputs to the environment.

Sampling

Samples should be collected in pre-sterilized glass bottles or in rubber syringes. Bottles containing thiosulphate may be used **but prior consultation with laboratory staff is required.** Samples should be packed in ice but should not be frozen.

Sulphur oxidizer levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, but not exceeding 48 hours.

Method

Sulphur-oxidizing bacteria are determined by a Most Probable Number (MPN) method. Media most often used by the MOE are thioparus, thiooxidans and ferrooxidans broths. Incubation is under aerobic conditions at 28 ± 0.5°C for 21 days. The final result is the concentration of sulphur oxidizing bacteria. Further testing can be done upon request to determine if heterotrophic sulphur oxidizers are included in this population of bacteria.

Time of Analysis

The number of analyses performed per day will depend on the type of sample and the number of dilution steps required. Sulphur oxidizer results are available 22 days after sample submission. **Prior consultation with Microbiology staff is necessary before this parameter is requested.**

MICROBIOLOGICAL

NUISANCE ORGANISMS

Certain microorganisms, although not pathogenic or indicative of the presence of fecal material, may be related to a deterioration in water quality or may reduce the efficiency of the treatment of water or sewage. Foul tastes and odours, slimes, discoloration, bulking of activated sludge, and the clogging or corrosion of treatment or distribution systems may all result from the presence of one or more groups of microorganisms. Nuisance organisms include iron bacteria such as Gallionella, Leptothrix, Sphaerotilus and Crenothrix, the sulphate reducing bacteria of the genera Desulfovibrio and Desulfotomaculum, the sulphur oxidizer Beggiatoa, actinomycetes, algae and fungi.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added). Samples should be packed in ice but should not be frozen. The sample should contain material representative of the problem such as slimes or filamentous growth collected aseptically.

Nuisance organism levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within 24 hours, but not exceeding 48 hours.

Method

With the exception of the sulphate reducing bacteria and fungi, identification is generally based upon direct microscopic observation of the sample. Morphological characteristics are generally used to identify the organism(s) present. The sulphate reducers may be cultivated and their presence revealed by the detection of metabolic byproducts. Fungi may also be cultivated and later identified by microscopic observation of their morphological characteristics.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of parameters requested and the incubation periods (if necessary) for each parameter. Results of microscopic analyses are available two days after sample submission, however, because the method and number of analyses vary with the sample problem, some analyses may take one month to complete. **Consultation with Microbiology staff is necessary prior to sample collection.**

MOISTURE CONTENT

The moisture content may be determined on solid or semi-solid samples such as soils, sediments and sludges. On soil and sediment samples, the percent moisture is required for the conversion of results reported as percent dry weight to percent wet weight. For sludge samples, the test is performed as a routine process control at the treatment plant. The test results, while traditionally reported as percent moisture are in fact a measurement of all volatile material in the sample. The test detects the loss of that fraction of the water content which will evaporate in 16 hours at 103°C as well as any other constituents which volatilize under the same conditions. The result reported therefore is frequently higher than the actual moisture content of the sample.

Sampling

Sediment samples should be collected in wide-mouth glass bottles with moisture-tight PVC closures. Bottles should not be filled to more than 70% capacity to allow space for gas generation. Soil and vegetation samples may be collected in glass pomade jars with moisture tight caps. Samples should be tested within 2 or 3 days of collection and stored under refrigeration. This is very important for sludges because of their high bacterial activity.

Method

A weighed sample is heated to 103°C to drive off moisture. The weight loss on heating is reported as a percent fraction of the initial weight taken. Other volatile materials in the samples may be measured along with the moisture. There may be a weight loss in sludges due to decomposing organic materials.

Time of Analysis

Approximately 30 samples may be analyzed per 24 hour period.

MOLYBDENUM

Molybdenum is found in nature as the minerals molybdenite, wulfenite, and molybdenum ochre. Its industrial applications include the manufacture of steel alloys, glass, ceramics, pigments, lacquers, paints, electrical wiring, and fertilizer. It is also used as a catalyst in certain chemical processes. Its presence in natural waters is likely the result of industrial discharges and agricultural run off as well as weathering processes.

In plants, molybdenum is believed to have an important influence on the ascorbic acid (vitamin C) equilibrium. It is essential to the nutrition of leguminous crops and all other nitrogen-fixing organisms. In animals, the function of molybdenum is not well understood but it appears to behave much like copper. It is known to be a constituent of certain flavoprotein enzymes which have essential metabolic functions.

Of all the industrial heavy metals, molybdenum is one of the least toxic. Normal soils contain between 3 - 5 mg/L. Isolated instances of molybdenum poisoning are generally associated with cattle grazing in pastures where the soil contains elevated levels of molybdenum (20 - 100 mg/l). This condition in cattle is commonly known as "teart" and is characterized by severe diarrhea, anemia, and harsh discolored fur.

Molybdenum is rarely encountered in domestic water supplies and no limit has been specified for its maximum acceptable concentration. Levels should not exceed 0.010 mg/L for irrigation waters used continuously on all soil.

Sampling

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per liter of sample. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested with an appropriate acid mixture and analyzed by atomic absorption. Sewage samples are analyzed by Inductively Coupled Plasma (ICP) atomic emission.

Time of Analysis

Approximately 50 molybdenum analyses can be performed per day.

✓

NICKEL

Nickel is more abundant in Ontario than anywhere else in the world. It is found primarily as the sulphide minerals pentlandite and pyrrhotite, as well as the arsenic mineral kupfernickel. Nickel has numerous industrial applications; the most important of which is the manufacture of high quality heat and corrosion resistant steel. Electroplating processes which utilize nickel are also widespread. Levels in natural waters are very low; therefore, if high nickel levels are found, they are likely the result of an industrial discharge or mine drainage. The median freshwater concentration of nickel in North American Rivers is 0.10 mg/L. LC

Nickel and its salts have generally proven to be non-toxic to man even at very high levels. Nickel sulphate and nickel bromide have even been used therapeutically for headache, neuralgia, and insomnia. However, nickel carbonyl is suspected of being carcinogenic although confirmatory evidence is not well established. Contact with the metal can cause a severe form of dermatitis. The element appears to be biologically active, activating several enzyme systems, and appearing in high concentrations in ribonucleic acid. Toxicity to plants and invertebrates varies with the species. Acute nickel toxicity to fish is generally less than that of copper or zinc. The 48-hour LC_{50} for rainbow trout is 32 mg/L while it is 0.750 mg/L for copper and 4 mg/L for zinc. Concentrations of nickel in an unfiltered sample should not exceed 0.025 mg/L to protect aquatic life. Irrigation waters which are used continuously on all soil should not contain more than 0.20 mg/L. source

Sampling

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per liter of sample. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Samples are digested in the appropriate acid mixture and analyzed. Sewage, sludge and Hi-Vol samples are analyzed by Inductively Coupled Plasma (ICP) atomic emission. All other samples are analyzed by atomic absorption.

Time of Analysis

Approximately 50 nickel analyses can be performed per day.

NITROGEN - AMMONIA AND AMMONIUM

Ammonia nitrogen is commonly encountered in domestic wastewater and industrial effluents. It is also found as a biochemical reduction product in ground water. Other sources include fertilizers, cleaning compounds and atmospheric precipitation.

Ammonia nitrogen may be toxic to fish because it reduces the oxygen carrying capacity of the blood. Ammonia toxicity is related to the amount of un-ionized ammonia and is dependent on pH, temperature and dissolved oxygen. Ammonia also exerts a high oxygen demand when converted to nitrite and nitrate, and interferes with water treatment procedures by reacting with chlorine to form chloramines. The latter reaction greatly reduces the effectiveness of the chlorination process. It is also objectionable because its nutritive properties promote the excessive growth of algae and other aquatic plants. Ammonia is rarely found in concentrations high enough to be harmful to humans. Concentrations in surface waters should not exceed 0.02 mg/L of un-ionized ammonia for the protection of aquatic organisms.

Ammonia results are of great value in interpreting the effectiveness of the waste stabilization process at sewage treatment plants. The relative abundance of ammonia in the treated water is an indication of how far treatment has progressed.

Ammonia is rather short lived in surface waters; therefore, it may not reveal the complete extent of pollution. Results must be interpreted with full allowance for its perishability, both in situ and in the sample following collection.

Sampling

Samples should be collected in PET plastic containers. To obtain reliable results, samples should be refrigerated and shipped to the laboratory as rapidly as possible. Samples may be frozen.

At room temperature bacteria can convert organic nitrogen to ammonia, and ammonia to nitrite. These reactions are accelerated for domestic wastes due to the abundance of bacteria.

Method

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system with nitroprusside as a catalyst, using a continuous - flow colorimetric system. Water extracts are used for soils and sediments. High levels of manganese, cobalt, iron, trivalent chromium, and calcium are known to produce interferences. Ammonia results are reported in mg/L of nitrogen. Ammonia in soils and sediments is reported as mg/g.

Time of Analysis

Approximately 200 ammonia analyses on aqueous samples may be performed daily.

NITROGEN - NITRATE

Nitrate is formed via the oxidation of nitrite by autotrophic nitrifying bacteria or through combustion and industrial processes, and represents the most highly oxidized form of nitrogen in the nitrogen cycle. It is generally found at trace levels in all surface waters but may become very high in ground water as a result of soil leaching. Potential sources of nitrates in water arise from the use of fertilizers, nitrogen fixation of micro-organisms and plants, decomposition of sewage wastes, leaching from soils and rocks and acidic precipitation. Nitrate provides an alternate source of oxygen when the dissolved oxygen has been depleted.

Nitrate results are of value in interpreting the effectiveness of the waste stabilization process at sewage treatment plants. Its abundance in treated water is an indication of how far treatment has progressed. Biochemical reduction of nitrate may frequently result in the production of nitrogen gas rather than ammonia (denitrification). This reaction is undesirable during the activated sludge process since the nitrogen gas tends to buoy the sludge and thereby inhibit digestion.

Nitrates are objectionable because their nutritive properties promote the excessive growth of algae and other aquatic plants. Although nitrates are considered non-toxic to adults, high levels in domestic water supplies contribute to a condition known as infant methemoglobinemia in which the oxygen carrying capacity of the blood is inhibited. The maximum acceptable level for domestic water supplies in Ontario is 10 mg/L of nitrate plus nitrite, reported as nitrogen if the water is to be used for infant feeding. Livestock water should not contain more than 100 mg/L of nitrate plus nitrite, reported as nitrogen.

Sampling

PET plastic containers are acceptable. To obtain reliable results, samples should be refrigerated and shipped to the laboratory as rapidly as possible. Samples may be frozen. Soil samples should be collected in pomade jars. Airborne nitrates are collected on glass fibre Hi-Vol filters. At room temperature bacteria can convert organic nitrogen to ammonia, and ammonia to nitrite. These reactions are accelerated in domestic wastes due to the abundance of bacteria.

Method

For precipitation samples, Hi-Vol filters, dustfalls and other aqueous extracts, nitrate is determined by ion chromatography. For other aqueous samples, nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 37°C, by hydrazine sulphate with copper as a catalyst. Colorimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N-(1-napthyl)ethylenediaminedihydrochloride. The analysis is performed on an AutoAnalyzer continuous - flow colorimetric system. Solid samples undergo extraction with water or potassium chloride before colorimetric analysis. Nitrate is calculated, and is the difference between the combined value for nitrate plus nitrite and the value for nitrite. High levels of copper, iron, calcium, magnesium and various oxidizing agents are known to interfere with the analyses.

Time of Analysis

Approximately 80-100 nitrate analyses can be performed per day by Ion Chromatography.

Approximately 200 nitrate analyses can be performed per day colorimetrically on aqueous samples.

NITROGEN - NITRITE

Nitrite is present in natural waters as an intermediary in the oxidation of ammonia by autotrophic nitrifying bacteria or in the anaerobic reduction of nitrate. In either case, its presence is short lived with the concentration seldom exceeding 1.0 mg/L as nitrogen. Higher concentrations are indicative of an industrial effluent. Waters containing nitrite are in the midst of rapid chemical changes.

Nitrite results may be used in interpreting the effectiveness of the waste stabilization processes at sewage treatment plants, but these results must be interpreted with full allowance for extreme perishability. The usefulness of nitrite results is questionable since it is highly unlikely that the concentration at the time of analysis represents the concentration at the time of sampling.

Nitrite is more toxic than nitrate. Domestic water should not contain more than 1.0 mg/L of nitrite as nitrogen. Livestock water should not contain more than 10.0 mg/L of nitrite as nitrogen.

Sampling

PET plastic containers should be used. To obtain reliable results, samples should be refrigerated and shipped to the laboratory as rapidly as possible. Samples may be frozen.

At room temperature bacteria can convert organic nitrogen to ammonia, and ammonia to nitrite. These reactions are accelerated for domestic wastes due to the abundance of bacteria.

Method

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide and N(1-naphthyl)-ethylenediamine dihydrochloride. Analyses are performed with a continuous - flow colorimetric, system. Solid samples undergo extraction with water before colorimetric analysis. High levels of free chlorine, nitrogen trichloride and colored substances may interfere. Results are reported as mg/L of N.

Time of Analysis

Approximately 200 nitrite analyses of aqueous samples can be performed per day.

NITROGEN - TOTAL KJELDAHL - IN AQUEOUS SAMPLES

Total Kjeldahl nitrogen is a measure of all the nitrogen in the trinegative state. It is primarily a measure of nitrogen present in the organic form (dissolved or particulate) such as proteins, amines and urea as well as nitrogen present as ammonia or ammonium compounds. It does **not** include nitrogen present as nitrite or nitrate. The organic nitrogen fraction can be estimated by taking the difference between the free ammonia and the total Kjeldahl nitrogen results.

Kjeldahl nitrogen results are of great value in interpreting the effectiveness of the waste stabilization process at sewage treatment plants. Treatment follows a natural sequence of biochemical reactions in which nitrogen components are successively oxidized from organic nitrogen to ammonia, nitrite and then nitrate and nitrogen gas. The relative abundance of each of the organic forms in the effluent is an indication of how far the treatment has progressed.

Sewage and industrial waste treatment effluents and runoff from agricultural areas, where fertilizers have been used, are the primary sources of TKN besides the natural nitrogen fixation and anaerobic nitrate reduction processes. There are no specific guidelines for total Kjeldahl nitrogen in drinking water although organic nitrogen levels should not exceed 0.15 mg/L.

Sampling

PET plastic containers should be used. To obtain reliable results, samples should be refrigerated and shipped to the laboratory as rapidly as possible. Samples may be frozen.

At room temperature bacteria can convert organic nitrogen to ammonia, and ammonia to nitrite. These reactions are accelerated in domestic wastes with abundant bacteria.

Method

Samples are pre-treated by digestion with sulphuric acid, mercuric oxide and potassium sulphate and then analyzed by the alkaline phenol hypochlorite method adapted to a continuous - flow colorimetric system. High levels of manganese, cobalt, iron, trivalent chromium and calcium are known to cause interferences. Kjeldahl nitrogen results are reported in mg/L of nitrogen.

Time of Analysis

Approximately 200 samples may be analyzed per day.

NITROGEN - TOTAL KJELDAHL - IN SOIL, SEDIMENT BIOMATERIAL AND VEGETATION

Total Kjeldahl nitrogen is a measure of all the nitrogen in the trinegative state. It is primarily a measure of nitrogen present in the organic form (soluble or particulate) such as proteins, amines and urea as well as nitrogen present as free ammonia. It does **not** include nitrogen present as nitrite or nitrate. The organic nitrogen fraction can be estimated by taking the difference between the free ammonia and the total Kjeldahl nitrogen results.

Kjeldahl nitrogen results are used to determine nutrient balance and uptake in aquatic vegetation, and to monitor the metal:nitrogen ratio in soils that have been used for municipal sludge disposal. It may also be used to assess damage as a result of industrial pollution on terrestrial vegetation.

The natural breakdown of organic material and the application of municipal sludge to agricultural soils are two primary sources of total Kjeldahl nitrogen.

Sampling

Kjeldahl nitrogen is slowly converted to other nitrogen compounds by bacterial activity in the presence of moisture.

Soil and vegetation samples should be collected in pomade jars and plastic bags respectively and both should be refrigerated and dried as soon as possible. Biomaterials should be kept frozen until ready for analysis.

Method

Solid samples are pre-treated by digestion with sulfuric acid and potassium persulfate. Analysis is accomplished using an automated alkaline phenol hypochlorite colorimetric procedure.

Results are reported as mg/g as N dry weight.

Time of Analysis

Approximately 120 samples may be analyzed per 5 day week.

CARBAMATE AND PHENYL UREA TYPE PESTICIDES

This group includes herbicides, fungicides and insecticides listed below. Carbamate herbicides are frequently used as pre-emergent treatment for grass seedlings and weed control in vegetable crops.

Carbamate insecticides behave similarly to organo-phosphorus insecticides, in that they depress cholinesterase activity and affect nerve transmission. Carbofuran levels should not exceed 100 µg/L in raw or livestock water supplies. It has been rated as a moderate ecological hazard. Benomyl and Captan are fungicides. Benomyl is very toxic to fish and invertebrates. The 48-hour LC-50 to fish is less than 10 mg/L. Carbamate insecticides and herbicides should not be found in livestock watering waters at levels exceeding 0.1 mg/L.

Pesticides included in the scan

BUX
BARBAN
BENOMYL
BUTYLATE (SUTAN)
AMINOCARB
CAPTAN
CARBOFURAN
CIPC
IPC
DIALLATE
EPTAM
PROPOXUR
METOXURON
METOBROMURON
MONVRON
MONOLINURON
SIDURON
CHLORTOLURON
DIFENOXURON
DIURON
FLUOMETHRON
LINURON

Sampling

Samples should be collected in baked, brown glass bottles with foil or Teflon lined caps. Bottles should be filled to the top, refrigerated and transported to the laboratory as quickly as possible.

Method

Samples undergo solvent extraction, clean-up with Florisil and analysis by (HPLC). High Performance Liquid Chromatography and ultraviolet detection. Detection limits are generally at 2 ppb in water.

Time of Analysis

Approximately 6 extractions can be performed per day, with a total routine analyses time of 2-4 days.

These are scan type workstations and little extra effort is required to report all parameters.

CHLOROPHENOL PESTICIDES

Chlorophenols exhibit germicidal and fungicidal properties and are used as biocides, fungicides and wood preservatives. In general, their effectiveness increases with the amount of chlorine substitution. Chlorophenols enter the environment through agricultural runoff and pulp and paper wastes.

Pentachlorophenol is used primarily as a wood and starch preservative but can be used also as an insecticide for termites, a pre-harvest defoliant and as a general herbicide. The trichlorophenols are often found as impurities in pentachlorophenol. The 2,4,5-compound is used as a fungicide and a bactericide. The 2,4,6-compound is used as a fungicide, a bactericide and a preservative.

Chlorophenols are extremely toxic to fish with a 96-hour LC_{50} for fish of less than 1 mg/L. Chlorophenols may also cause taste and odour problems in drinking waters.

At present there are no specified control limits for chlorophenols.

Sampling

Samples should be collected in 1 L baked brown glass bottles with Teflon lined caps. If absolutely necessary, foil lined caps will be accepted. Bottles should be filled to the top. Samples should be refrigerated and transferred to the laboratory as quickly as possible.

Method

Samples undergo solvent extraction, evaporation, derivatisation, clean-up with Florisil and analysis by electron capture gas chromatography.

At present this test measures 2,4,6-trichlorophenol, 2,4,5-trichlorophenol, 2,3,4-trichlorophenol, 2,3,5,6-tetrachlorophenol, 2,3,4,5-tetrachlorophenol and pentachlorophenol (PCP).

Time of Analysis

Approximately 12 extractions can be performed per day, with a total analysis time of 3-5 days. This is a scan type workstation and little extra effort is required to report all parameters.

dissociate to chlorophenate ions in water and the degree of dissociation depends on both the pH of the water and on the dissociation constant of the specific CP isomer. The degree of dissociation increases with pH. The lower CP's do not dissociate as readily as the higher CP's at normal environmental pH. Because the dissociated forms of CP's are considerably less toxic than the undissociated forms, increasing pH tends to decrease CP toxicity.

Although few studies have examined the effect of water hardness on CP toxicity, the available data indicate that toxicity is not appreciably diminished by increasing hardness (e.g., Birge et al. 1979).

Recommended Criteria

The surface water quality criteria recommended for the protection of aquatic biota are summarized in Table 3. These values are based on acute and chronic toxicity data and on threshold concentrations for the tainting of fish flesh. All values are derived to protect the most sensitive Ontario species from both toxicity (acute and chronic) and flavour impairment.

The most sensitive response of aquatic biota to MCP and DCP is flavour impairment. Recommended criteria are, therefore, less than the lowest tainting thresholds for these lower CP's. The conventional approach of using acute toxicity data and standard application factors to derive criteria would not protect aquatic biota from flavour impairment.

The recommended criterion for TCP is based on the lowest acute LC_{50} value multiplied by an application factor of 0.05 for non-persistent contaminants. Non-persistent toxic contaminants are those having half-lives in surface water environments of less than eight weeks, as stipulated by the International Joint Commission in the Great Lakes Water Quality Agreement of 1978.

Recommended criteria for TTCP and PCP are based on the lowest acute LC_{50} values multiplied by an application factor of 0.01 for persistent contaminants. Persistent toxic contaminants, as defined by the International Joint Commission, have half-lives of greater than eight weeks in surface water environments.

TABLE 3: RECOMMENDED SURFACE WATER CRITERIA FOR CHLOROPHENOLS

Chlorophenol	Recommended Criterion[1] (ug/L)	Basis
Monochlorophenols cP	7	flavour impairment
Dichlorophenols DCP	0.2	flavour impairment
Trichlorophenols TCP	18	acute toxicity, non-persistent
Tetrachlorophenols TTCP	1	acute toxicity, persistent
Pentachlorophenol	0.5	acute toxicity, persistent

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[1] Criteria are applicable to all isomers within each chlorinated class.

ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS

The organochlorine pesticides (OC's) form a class of compounds which includes widely used insecticides such as DDT. Polychlorinated biphenyls (PCB's) are not used as pesticides but have many industrial uses.

OC's can be classified as halogen derivatives of alicyclic hydrocarbons (BHC, lindane, heptachlor, aldrin, dieldrin, endrin) or aromatic hydrocarbons (DDT, DDD, DDE, endosulfan, HCB, methoxychlor).

The possibility of environmental contamination by organochlorine pesticides and polychlorinated biphenyls is of particular concern in view of their persistence. The stability of these products allows them to persist in the environment indefinitely creating a special hazard to wildlife due to the capability of many organisms to concentrate these compounds. This means that low levels (ng/L) of OC's and PCB's in water can be biomagnified up the food chain so that fish and fish-eating birds may contain mg/kg levels of these products. PCB's and DDT have been implicated in egg shell thinning in wild birds, endangering survival of the species.

Generally, OC's are resistant to degradation and oxidation, are relatively insoluble in water, have low volatility, and are lipophilic. Similarly, PCB's are inert, have low volatility and are practically insoluble in water (aqueous solubility varies with the chlorine content of a PCB fraction, from 40-200 µg/L), however, they are also resistant to acids, alkalis and high temperatures. PCB's are used in protective coatings, plasticizers, water proofing compounds, asphalts, inks, casting waxes, adhesives and as dielectrics, hydraulic fluids, grinding fluids and high pressure lubricants. Generally, the acute toxicity of organochlorine pesticides is low except for aldrin, endrin, toxaphene and endosulfan where the LD_{50} is in the 12 - 50 mg/kg range. Maximum concentrations for the protection of aquatic life in unfiltered samples are: 0.001 µg/L for Aldrin, Dieldrin and Heptachlor; 0.003 µg/L for DDT and metabolites; 0.002 µg/L for Endrin and 0.01 µg/L for Lindane. The edible portion of fish should not exceed 0.3 µg/g of aldrin, dieldrin, endrin, heptachlor or lindane for the protection of human consumers of fish. Drinking water levels are 17.0 µg/L for Aldrin and Dieldrin, 18.0 µg/L for Heptachlor, 1 µg/L for Endrin, 42 µg/L for DDT's and 56 µg/L for Lindane.

PCB's should not exceed 0.001 µg/L in an unfiltered sample to protect aquatic life and 3 µg/L in drinking water.

Sampling

Water, oil (PCB only analysis available), soil, and vegetation samples should be collected in baked brown glass bottles or jars with foil or Teflon lined caps. Plastic bottles should not be used as plasticizers may leach into the sample and interfere with the analysis. All these samples should be refrigerated immediately at 2 - 5°C. Fish samples should be wrapped in solvent rinsed aluminum foil, frozen and stored at - 20°C.

Method

A sample is solvent extracted, dried, concentrated and cleaned up to remove interferences. The clean extract is examined by Gas Chromatography using Electron Capture detector. For water samples, if no PCB's are present, pesticides are determined directly on the clean extract fractions by standard calibration and peak height quantitation. PCB's in fish, soil, sediment and water samples are separated from organochlorine pesticides by Florisil column chromatography, enabling the determination of each separately as above.

Time of Analysis

Approximately 15-20 extractions may be performed per day, with a total routine analysis time of 5 days. This is a scan type workstation and little extra effort is required to report all parameters.

ORGANOPHOSPHATE INSECTICIDES

Because of their low persistency and high effectiveness organophosphate insecticides are now used world wide. Hundreds of organophosphate compounds have found application as pesticides, mostly insecticides. In general they are esters of phosphoric, thiophosphoric or dithiophosphoric acids. There are wide variations in chemical structures, water solubility, volatility, stability and toxicity. Their low persistency is the result of chemical degradation or metabolism by living organisms to simple organic or inorganic phosphates.

Organophosphate insecticides function by blocking the activity of the enzyme cholinesterase and thereby interfering with a major life function, neural transmission in mammals and arthropods.

Mammalian toxicities range from 0.1 to 1000 mg/kg. (LD_{50}).

Levels should never exceed 100 µg/L in raw water supplies or in livestock water supplies. Levels which can be harmful to aquatic life vary from 0.001 µg/L for dichlorvos to 0.08 µg/L for diazinon. The Environmental Protection Service has given the following ratings for Ecological Hazards: Extreme - phorate and dursban; High - diazinon, malathion and ethion; Moderate - dichlorvos and parathion.

Sampling

Aqueous samples should be collected in baked brown glass bottles with Teflon lined caps. If absolutely necessary, foil lined caps will be accepted. Bottles should be filled to the mark. Solid samples should be collected in pomade jars with foil lined caps. All samples should be refrigerated and transferred to the laboratory as quickly as possible. Some organophosphates eg. diazinon and methyltrithion should be transported to the laboratory within 24 hours, or sooner if possible since they are very unstable in aqueous solution.

Method

Samples undergo solvent extraction, Florisil clean-up and analysis by thermionic specific detector gas chromatography.

This test now detects dichlorvos, phorate, diazinon, parathion, methyl-parathion, malathion, ethion, methyltrithion, Dursban, Reldan, Guthion, mevinphos, and ronnel.

Time of Analysis

Approximately 20 samples can be extracted per day, with a total analyses time of 3 days.

Phenoxyacetic acid herbicides (2,4-D type) are widely used for selective weed control in crops that are comparatively resistant to these chemicals.

Potential problems may arise when agricultural run-off waters containing 2,4-D type residues are used to irrigate a crop which is susceptible to these residues, thus causing serious damage. These materials can also impart severe taste and odour problems to water supplies at low milligram per liter levels. Part of the taste problems arise from the ability of the herbicides to decompose to chlorophenols.

The toxicity of 2,4,-D type herbicides to mammals is relatively low. However, highly toxic impurities (such as dioxins) may be produced as by- products in the manufacture of these herbicides.

The maximum permissible levels in raw waters for 2,4-D, 2,4-DB, 2,4,5-T, 2,4-DP, silvex and MCPA are 100 µg/L. Levels for protection to aquatic life are generally not specified, but 2,4-D and silvex should not exceed 4.0 and 2.4 µg/L respectively. The Environmental Protection Service has designated picloram as high, 2,4-D, 2,4,5-T and silvex as moderate and 2,4-DB and 2,4-DP as slight ecological hazards.

Sampling

Aqueous samples should be collected in baked brown glass bottles with Teflon lined caps. Solid samples should be collected in specially cleaned 500 mL brown wide mouth jars with foil lined caps. All samples should be refrigerated and sent to the laboratory as quickly as possible.

Method

All samples undergo solvent extraction, evaporation, derivatisation, clean-up with Florisil and analysis by electron capture gas chromatography.

Time of Analysis

Approximately 8 samples can be extracted per day, with total analysis time 2-3 days.

TRIAZINE HERBICIDES

Triazine herbicides are widely used in modern agriculture as selective weed killers. This test presently detects the following substances: atrazine, simazine, propazine, bladex, prometrone, ametryne, prometryne, sencor and atratone.

The maximum permissible level for triazine herbicides is 100 µg/L in the raw water supply. However, recommended levels for raw waters are 7.5 µg/L for Atrazine, 75 µg/L for Simazine and 16 µg/L for Propazine. Simazine levels should not exceed 10 µg/L in an unfiltered sample to protect aquatic life. Levels for other compounds in this group are not specified. Irrigation waters should not have residues of triazines in excess of 0.05 µg/L to avoid crop damage.

Bladex has been designated as a high ecological hazard, promatone and simazine as moderate hazards and atrazine as a slight hazard by the Environmental Protection Service.

Sampling

Aqueous samples should be collected in baked brown glass bottles with Teflon lined caps. If absolutely necessary, foil lined caps will be accepted. Bottles should be filled to the top. Solid samples should be collected in baked 250 mL amber jars with foil lined lids. All samples should be refrigerated and transported to the laboratory as quickly as possible.

Method

Samples undergo solvent extraction, evaporation, clean-up with alumina and analysis by gas chromatography using a nitrogen/phosphorus specific detection system.

Time of Analysis

Approximately 8 extractions can be performed per day, with total analysis times of 2-5 days.

PARTICLE SIZE DISTRIBUTION

The solid phase of soil and sediment is comprised of structurally diverse organic and inorganic particles which may be crystalline or amorphous and which vary greatly in size and shape. The measurement is generally restricted to the inorganic component. Although these particles are generally found as aggregates, they can be separated by chemical and mechanical means.

Many soil textural classification systems are based on particle size measurements, however, the variety of procedures used makes comparisons between classification schemes difficult. Particle size is a significant controlling factor for porosity, permeability, water holding capacity, leaching potential, cation exchange, weathering behaviour and several other parameters. Textural measurements on bottom and suspended sediments may be used to determine relationships between water quality, flora and fauna populations and bottom sediment characteristcs. The fractionation of soils and sediments into various size classes can be useful for a correlation of pollutant concentration with texture.

Sampling

Samples should be collected in glass or plastic bottles.

Method

Method A involves sieve analysis to separate the sand fraction. Method B employs a hydrometer to measure changes in density of a suspension in a sedimentation cylinder. Density changes can be related to the settling velocity of the particles as determined by Stokes Law. Method C employs the pipette method in which aliquots are withdrawn from a sedimentation cylinder at a predetermined time. This aliquot is then dried and weighed. This method requires more time and care than method B. Method D involves the use of a Sedimentation balance. This method allows more accurate measurement of the silt fraction than Methods B or C. Method E involves analysis by Microtrac Particle Size Analyzers. This is the method used routinely for soil and sediment analysis. Methods A to D are used only on special request for analysis of Terrestrial Effects soil samples. **Please contact supervisor of Soil Unit first.**

Time of Analysis

Method A - 1 day per sample if pretreatment is required.
Method B - 10 samples per 24 hours.
Method C - 10 samples per 24 hours.
Method D - 1 sample per 24 hours.
Method E - 5 samples per 24 hours.

PETROLEUM HYDROCARBONS

Petroleum hydrocarbons include gasolines, aviation fuels, organic cleaners, thinners, kerosenes, stove oils, home heating fuels and transformer oils. Many of these substances may impart objectionable tastes and odours to domestic water supplies, even at low levels. They are also known to increase the oxygen depletion rate in receiving waters and will hinder oxygen exchange with the atmosphere by forming slicks.

Petroleum hydrocarbons are generally found as complex mixtures of organic compounds. Thus, identifications may be reported as a class or type of material rather than as a specific substance. Decomposition of the sample may alter part of the chromatogram which is used for quantitative purposes. Although the results are considered to be only semi-quantitative they may give an indication of the magnitude of pollution present. Qualitative results are obtained by direct comparison with known standards. The source of the contaminant may be identified by comparing the chromatogram of the extracted pollutant with those of samples collected from storage tanks in the spill area. Chromatograms are generally assessed qualitatively and semi-quantitatively for liquid samples and qualitatively for soil samples. To protect fresh water fish, concentrations should not exceed 180 µL/L for bunker oil, 120 µL/L for used crankcase oil, 45 µL/L for jet aviation fuel and crude oil slicks, 15 µL/L for diesel oil and 9 µL/L for automobile gasoline.

Sampling

Open waters should be sampled at the surface as this is where petroleum hydrocarbons will collect. If closed plumbing systems are to be sampled, taps should **not** be opened for several hours prior to collection so that contaminants may build up. Tap sieves and toilet tanks are frequent areas where contaminants collect. Samples of the suspected contaminant source should be submitted with the contaminated water whenever possible.

One full 1L glass bottle, clearly labelled "PETROLEUM HYDROCARBONS" should be submitted for this test exclusively. Bottle caps must be aluminum foil or Teflon lined as any rubber or plastic caps may release organic compounds. Containers must be tightly capped and refrigerated to prevent the loss of volatile components. Solid samples should be stored in pomade jars with a foil lined lid, and kept refrigerated. Petroleum hydrocarbons are susceptible to bacterial attack; therefore immediate delivery to the laboratory is essential.

Method

An extraction is performed using an organic solvent such as n-hexane and analysis is accomplished by gas chromatography.

Time of Analysis

Approximately 5 to 10 petroleum hydrocarbon determinations can be performed per day.

how about B.T.X. - the WRB has water quality objectives for the attached list of aromatic cpds + related cpds.

TABLE 2

Criteria Summary and Factors Influencing Criteria Recommendations

Compound	Type	Criterion Maximum	Criterion 24-Hr. Avg.	Critical Factors Influencing Development of Criterion
Benzene	Recommended	50 ug/L	25 ug/L	Abnormal metabolism in fish embryos caused by sublethal levels; compound is a known carcinogen
Toluene	Recommended	500 ug/L	250 ug/L	Lowest 96-hr LC_{50} of most sensitive aquatic species tested
Xylene	Recommended	100 ug/L	50 ug/L	Avoidance by juvenile Coho Salmon
Ethyl-benzene	Preliminary	1400 ug/L	700 ug/L	Lowest 96-hr LC_{50} of most sensitive aquatic species tested
Styrene	Preliminary	200 ug/L	100 ug/L	Potential for bioconcentration potential for flesh tainting
Aniline	Recommended	40 ug/L	20 ug/L	Lowest 96-hr LC_{50} for a sensitive aquatic species
Nitro-benzene	Preliminary	1000 ug/L	500 ug/L	48-hr "no discernible effect concentration" for most sensitive aquatic species tested
Dinitro-benzene	Preliminary	60 ug/L	30 ug/L	Lowest 96-hr LC_{50} for a sensitive aquatic species

SUMMARY

In formulating the following Provincial Water Quality Objectives for chlorinated benzenes their production and uses, chemical and physical characteristics, interactions and persistence in the aquatic environment, and toxicity to aquatic biota have been considered.

Quantitative Structure-Activity Relationships (QSAR), based on aquatic toxicity data and hydrophobicity (as measured by octanol-water partition coefficients, log K_{ow}), were used to produce estimated toxicity data where experimental data were scanty or absent. As well, QSAR methodology was used to interpret and generally rationalize the development of objectives. This allowed objectives to be developed for all chlorinated benzene compounds and their isomers.

Although objectives are usually considered to be primarily for the protection of aquatic life, it is not reasonable to propose objectives that might impair other uses. Since there is sufficient information on bioaccumulation hazard to suggest that other uses might be affected at levels judged acceptable in terms of toxicity to aquatic biota alone, consideration of bioaccumulation hazard has been incorporated in the objectives development process.

A summary of the proposed Provincial Water Quality Objectives for chlorinated benzenes follows.

		Objective (µg/L)
Monochlorobenzene		15
Dichlorobenzene	1,2	2.5
	1,3	2.5
	1,4	4.0
Trichlorobenzene	1,2,3	0.90
	1,2,4	0.50
	1,3,5	0.65
Tetrachlorobenzene	1,2,3,4	0.10
	1,2,3,5	0.10
	1,2,4,5	0.15
Pentachlorobenzene		0.030
Hexachlorobenzene		0.0065

PETROLEUM HYDROCARBONS
GASOLINE AND FUEL OIL TRACING WITH SYNTHETIC MUSKS

Leakage of gasoline or fuel oil from underground storage tanks is one of the most frequent causes of groundwater contamination in Ontario.

For gasoline, the nearest service station is always suspect. Even if there is only one gas station in the area, two or three tanks may be leaking. Pressure tests do not always work and it is difficult to distinguish between different grades of gasoline after it has passed through soil and water. Since excavation of the suspected tank is prohibitively expensive for inspection, a reliable indicator is required before such an operation is undertaken. When there are two or more service stations at an intersection, the problem can be very complex.

The application of Musk tracers to such problems was developed by the Laboratory to help field investigations. Special musk compounds used in perfumery were chosen as tracers because these are considered non-toxic and are available as high purity chemicals. They have very low analytical detection limits and have no damaging effect on internal combustion engines. The compounds used are musk xylol, musk ambrette, musk ketone, musk tibetene, and moskene (in order of increasing price). Musks are dissolved in toluene (250 g in 1 L), diluted with 20-40 L of gasoline and added to the suspected tank to give a 50 mg/L (ppm) final musk concentration in gasoline. Up to five tanks can be tagged simultaneously with different musks.

Test holes or affected wells are monitored for gasoline and musk content. This technique has been applied successfully on several occassions.

Sampling

Aqueous samples should be collected in 1 L glass bottles with foil lined caps. When gasoline contamination is visible, a few mL of gasoline sample is sufficient for musk analysis.

Method

Samples are solvent extracted and analysed by gas chromatography using a nitrogen specific detector.

NOTE: Analysis for musks is not routine and special arrangements must be made. **Discuss the problem in advance with laboratory personnel.** Analysis of fuel oil for musks is more complex but can be handled on a project basis.

pH

pH is defined as the negative logarithm of the hydrogen ion activity, but in the case of natural water samples, the hydrogen ion activity closely approximates the hydrogen ion concentration in moles per liter. As the pH scale is logarithmic, a change of pH by one unit means a tenfold change in the hydrogen ion concentration. Rivers and lakes which have not been polluted by acid or alkaline wastes range in pH from about 5 (acid peat bogs) to 8.5 (chalk streams and marl lakes). In highly productive waters, photosynthetic activity can cause the overabundance of carbonate ion. This may also result in a natural pH close to 8.5.

Most surface waters are protected from fluctuations in pH by the natural buffering capability of the bicarbonate ion. Pollution by industrial wastes and acid precipitation, however, can upset this equilibrium and produce significant pH changes in the receiving water. A change in pH implies that changes in the alkalinity and acidity are also taking place. pH results are best interpreted in conjunction with the alkalinity and acidity.

pH is a highly perishable parameter because of its dependence on temperature, dissolved gas concentrations, and other chemical reactions. It should be measured in the field whenever possible. A laboratory pH measurement is not necessarily representative of the pH at the time of sampling.

Because both alkaline and acidic waters may cause eye irritation, the pH of the water used for recreational purposes should be within the range of 6.5 to 8.5.

Sampling

PET plastic containers should be filled to capacity so that no air bubbles remain once the samples are capped. For sludges, fill only two thirds. Soil samples should be collected in pomade jars. Do not refrigerate. Immediate delivery to the laboratory is essential.

Method

The pH is measured directly on a stirred aliquot of sample at room temperature. A high quality pH meter and electrode are used. The pH of soil and sediment samples is measured on a 0.01 M $CaCl_2$ or distilled water extract. The sample temperature, degree of electrode immersion and stirring rate should all be as constant as possible.

Time of Analysis

Approximately 100 pH analyses can be performed per day.

The reactive phosphorus content of an aqueous sample is that fraction which reacts with the reagents present to produce a positive colorimetric response. It is possible that some organic and particulate forms of phosphate can react similarly to dissolved orthophosphate under the analytical test conditions. The above terminology should remove the implication that the test measures only orthophosphate forms which are uncombined and in solution.

Phosphorus is an essential element for all life forms. Artificial inputs of phosphorus via wastewater and land run-off promote an overabundance of algae and aquatic plants which may impair water quality.

Phosphates are used in some water treatment methods, in commercial cleaning preparations, to treat boiler waters, and as fertilizer.

The usefulness of reactive phosphorus results is questionable because of its reactivity. Results must be interpreted with full allowance for perishability, both in situ and in the sample following collection.

Sampling

Use brand new PET plastic containers when sampling for filtered reactive phosphorus. Re-washed containers are likely to remain contaminated with a residual amount of phosphorus, regardless of how thoroughly they are washed. Reactive phosphorus is readily converted to other phosphorus forms biologically, so samples must be refrigerated or frozen immediately after collection and delivered to the laboratory as soon as possible.

Method

Orthophosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent. The analyses are performed on an AutoAnalyzer continuous - flow colorimetric system. High levels of iron (III), hexavalent chromium, arsenic (V), stannous ion, and some colored compounds are known to interfere. Results are reported as mg/L of phosphorus.

Time of Analysis

Approximately 200 analyses may be performed per day. Other soluble nutrients such as nitrite, nitrate and ammonia may be determined at the same time.

This outline includes two types of tests: Extractable Phosphorus and Total Phosphorus by dissolution.

Phosphorus occurs in nature in a variety of organic and inorganic forms. There is a continual process of uptake of inorganic forms, conversion to organically bound phosphorus and decomposition of these organic forms to oxidized inorganic forms.

Phosphorus is derived from leaching and weathering of igneous rocks and phosphorus minerals such as apatite, domestic sewage and fertilizers. Phosphorus in acidic soil and sediment is often associated with iron and aluminum while it is associated with calcium in alkaline materials. While most forms of phosphorus are biologically available, apatite phosphorus is not.

The extractable phosphorus tests for soil and sediment use one or a sequence of extractions to determine different forms of phosphorus, whereas the total phosphorus by dissolution uses only a one step digestion to dissolve the total amount of phosphorus present.

Sampling

Samples for extractable phosphorus in soil and sediments should be collected in pomade jars, and air-dried at the lab. Samples for total phosphorus can be air or oven dried.

Method

Extractable phosphorus

These analyses are performed by special request only. Samples are extracted sequentially with sodium hydroxide (for iron and aluminum phosphates), a sodium citrate-bicarbonate-dithionate solution (for occluded phosphates) and hydrochloric acid for calcium phosphate. Phosphorus in the various solutions is analyzed colorimetrically using an ammonium molybdate-stannous chloride method. Sodium bicarbonate is used as an extractant for "available" phosphorus in sewage sludge amended soils.

Total phosphorus by dissolution

All phosphorus is converted to the inorganic form by digestion in hot sulphuric acid with the addition of potassium persulphate. After elimination of interfering iron in soil and sediment digestates by precipitation and filtration, phosphorus is measured colorimetrically using an ammonium molybdate-stannous chloride AutoAnalyzer method.

Time of Analysis

Approximately 30 samples may be analyzed per day.

PHOSPHORUS - TOTAL

Phosphorus occurs in natural waters and wastewaters in several different combinations, which include orthophosphates, pyrophosphates, metaphosphates, polyphosphates and organically bound phosphates. Phosphorus is an essential element for all life forms. As all of the above compounds may eventually be used in metabolic processes, determination of the total phosphorus concentration is more relevant environmentally than the measurement of individual phosphorus compounds.

Phosphates are used in the manufacture of fertilizers as well as detergents. Polyphosphates are used in some public water supplies and industrial boiler systems as scale and corrosion inhibitors. Artificial inputs of phosphorus via wastewater and land run-off promote the over-abundance of algae and aquatic plants which impair water quality. Phosphorus results are used in assessing a water's potential for biological productivity as well as the efficiency of nutrient removal facilities at waste treatment plants. To avoid nuisance levels of algae in lakes, average total phosphorus concentrations for ice free periods should not exceed 20 µg/L. To protect against aesthetic deterioration, total phosphorus levels should not exceed 10 µg/L during ice free periods.

Sampling

Contamination is a significant problem in determining total phosphorus concentrations. Re-washed bottles may contain residues of phosphorus, hence single-use PET plastic containers are used. If using a re-washed container is unavoidable, it must be cleaned with 10% hydrochloric acid and thoroughly rinsed with distilled water prior to field use. Storage in the dark is recommended for total phosphorus to prevent algal growth on container walls. The sample may be refrigerated.

Method

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters kept at 200°C and 360°C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent. The analyses are performed on a continuous - flow colorimetric system. High levels of iron (III), arsenate, and hexavalent chromium are known to interfere. Interferences by colored compounds should be removed by the digestion procedure. Results are reported as mg/L of phosphorus.

Time of Analysis

Approximately 200 samples may be analyzed per day. Total Kjeldahl nitrogen may be performed simultaneously.

POTASSIUM

Potassium ranks seventh in the natural order of elemental abundance. It is highly soluble and present at trace levels in all natural surface waters, but very seldom exceeds 10 mg/L. Brackish or saline ground water, however, may contain potassium in excess of 100 mg/L. Potassium is generally found in the presence of sodium and when potassium is in greater abundance than sodium, an artificial source is likely. The sodium:potassium ratio in natural waters generally ranges from 2:1 to 10:1 with the proportion of sodium increasing with increasing concentrations.

Potassium is an essential nutrient for all life forms, especially plants, and is therefore a major component of commercial fertilizers. Despite the substantial amounts of potassium dispersed over the land as fertilizer, it seldom appears in any great quantity in the water system. Many soils have the ability to fix potassium and prevent its migration through leaching. It is non-toxic and represents no known health hazard unless at extremely high levels. No limits have been specified for the maximum acceptable concentration of potassium in domestic water supplies.

Sampling

PET plastic containers must be used for aqueous samples. Soil samples should be collected in pomade jars. Vegetation samples should be collected in perforated plastic bags and kept refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

River, precipitation and domestic water samples are analyzed using an automated atomic absorption technique. Cesium chloride is added to minimize partial ionization of potassium ions in the flame.

Soil and vegetation samples are analyzed using atomic absorption after ashing and solution in hydrochloric acid.

Potassium in vegetation samples is also measured by X-ray fluorescence. Results are reported as % dry weight of the sample (See Matrix elements in Vegetation).

Time of Analysis

Approximately 150 potassium analyses may be performed per day on aqueous samples and 50 per day on solid samples.

PHYTOPLANKTON

Phytoplankton (algae) are normal biological inhabitants of nearly all natural surface waters. As green plants containing chlorophyll they are actively involved in the photosynthetic process. They use the nutrients and minerals in the water and absorb carbon dioxide to grow and reproduce. Bacteria, protozoa and other microscopic animals feed on the algae and absorb the oxygen produced by them. Problems arise in the aquatic environment when the balance between the algae and animal organisms is upset, such as through the introduction of pollutants.

The general public is not usually aware of these minute algal organisms unless they become so abundant that collectively they form visible green masses or scums. They may accumulate on the shoreline and impair the use of that area for recreational or aesthetic pursuits. Algae may also enter water supply facilities where they may cause operational problems such as filter clogging or taste and odours. The quantity of algae capable of imparting chemical flavours (cucumber, grassy, musty, fishy etc.) in water may not be great but it can be sufficient to make the water undrinkable for the consumer.

The Ministry of the Environment has facilities for **identifying** most forms of freshwater algae, be it microscopic free-floating phytoplankton or larger attached forms that appear as visible scums in lakes, ponds, swimming pools, bird baths, roof tops and even moisture-laden lawns. **Quantitative** estimates can be performed on unconcentrated whole lake or raw water samples only. Biomass estimates of standing algae crops are reported as Areal Standard Units per millilitre (A.S.U./mL) for water supplies and as mm^3/L (g/m^3) for recreational or water quality assessments.

Sampling

Add 2 mL of acidified Lugols' iodine solution to 1 litre of sample for **quantitative** analysis of phytoplankton. Samples may be collected as surface grabs (1 foot below surface), as photic zone composites from in-lake stations or as raw water intake samples from drinking water supply sources. Please contact the Aquatic Biology laboratory prior to intiating any sampling programme by calling 235-5811.

Samples for algae **identification** only may be collected in 1 litre bottles if they are for the examination of microscopic forms such as taste and odour producing organisms. They should be submitted in duplicate (one live sample and one preserved sample). Samples for the identification of algal scums may be collected in 1 oz. vials or any small wide-mouth container (in the ratio of 1 part of algae to 10 parts water) and preserved.

Method

Samples for **quantitative** analyses are concentrated by sedimentation with Lugols' iodine solution in the laboratory to provide a 25 mL aliquot which is stored until time of analyses. Microscopic analyses is performed using 2-5 mL Utermohl sedimentation chambers in combination with inverted light microscopes at a magnification of 600X. Sufficient transects of the counting chamber are examined to provide statistically significant numbers and measurements of dominant taxa. Taxonomic identifications are made to the generic level and to the species level where possible or necessary.

Time of Analyses

Approximately 10 **quantitative** samples per day can be completed. Samples may be held to end of programme to facilitate identifications and trends in seasonal changes. **Identifications** may be completed within 48 hours.

SELENIUM

Selenium is naturally found in the form of metallic selenides, selenites, and selenates, and also as organic compounds derived from decayed plant material. Selenium is likely to be present in natural waters as the selenate anion SeO_4 or as organic selenium compounds which have been discharged in industral effluents. The elemental form is insoluble in water but it may be transported by being adsorbed onto particulate matter. Sediment material seems to act as sinks or reservoirs for selenium. While water which has seeped through coal and shale deposits may contain up to 0.3 mg/L most surface waters contain less than 0.001 mg/L. Its common industrial applications include the manufacture of rectifiers, photoelectric cells, red colored glass, pigments, paints, dyes and insecticides, as well as in the vulcanization of rubber as a supplement to sulphur.

Deficiency of selenium in the grass eaten by livestock may lead to white muscle disease. Selenium poisoning in mammals may be of the acute type, called "blind staggers", or the chronic type, called "alkali disease". In humans, acute toxicity may be characterized by nervousness, vomiting, cough, convulsions, abdominal pains, diahrrea and respiratory failure. Chronic effects include weakness, visual impairment, damage to the heart and liver, tooth decay, red tainting of fingers, teeth and hair, depression, and irritation of nose and throat. Claims of carcinogenic potential have yet to be proven.

Selenium may decrease the toxicity of cadmium and mercury.

The 96 hour LC_{50} values for zebra fish larvae indicate that selenates are less toxic than selenite salts by about a factor of 4. A lethal threshold value of 2.5 mg/L has been found for Daphnia sp. and Scenedesmus sp.

The maximum acceptable concentration of selenium for domestic water supplies in Ontario is 0.01 mg/L. Concentrations in an unfiltered sample should not exceed 0.1 mg/L to protect aquatic life. Fish with levels of 3 µg/g wet weight, should not be eaten often. Livestock waters should not contain more than 0.05 mg/L while irrigation waters should not contain more than 0.02 mg/L.

Sampling

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per liter of sample. Pomade jars are used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Sample aliquots are digested with oxidizing acid mixture. Hydrogen selenide gas is generated in acid solution by Sodium borohydride which is atomized in a heated quartz tube and measure by atomic absorption.

Time of Analysis

Approximately 50 samples can be analyzed per day.

SILICATES - REACTIVE

The element silicon ranks second to oxygen in natural abundance in the earth's crust and is present as silica or silicates in sand or quartz. In natural water silicates are generally found as monomers, polymeric chains and cyclic anions in both soluble and colloidal forms. Surface water levels are usually under 5 mg/L as Si but higher levels are not uncommon, especially in ground waters.

Silica and silicates are widely used in industry for the manufacture of glass, ceramics, abrasives and enamels and have been used in domestic water treatment as coagulants, corrosion inhibitors, and sequestering agents for iron. Their presence in boiler feed waters causes deposits of silica on steam-turbine blades, necessitating removal to concentrations as low as 0.1 mg/L in high pressure systems.

Silicates are essential to the growth of diatoms as well as many other aquatic organisms. No adverse physiological effects are known at concentrations generally found in natural or treated waters. Therefore, no maximum concentration has been specified for its presence in domestic water supplies.

Sampling

PET plastic containers must be used for aqueous samples requiring silicate analysis. Glass bottles should not be used since silicon is a major constituent of glass. Analyses should be completed as quickly as possible since biological activity can reduce the dissolved silicate concentrations in enriched natural samples. Vegetation samples should be collected in perforated plastic bags and kept refrigerated.

Method

Water samples are analyzed directly using a molybdate method with a continuous - flow colorimetric system. This method measures all forms of silicon which are hydrolyzable under the conditions of the test as the reactive silicate ion. Silica in the soluble colloidal and insoluble polymer forms generally won't react with the molybdic acid. High concentrations of iron, tannins, lignins, sulphides and phosphates may interfere; however, many of these can be overcome. Highly colored or turbid samples may have some interference. Results are reported as mg/L of silicon.

Vegetation samples are analyzed by X-ray fluorescence. Results are reported as % of dry weight. (See Matrix elements in Vegetation).

Time of Analysis

Approximately 200 silicate analyses can be performed per day on aqueous samples.

SILVER

Silver is found in nature as the native metal, but its chief source is the mineral argentite. Besides being relatively rare, most of its compounds are insoluble; therefore it is highly unlikely that silver would appear in a domestic water supply unless introduced by artificial means. Weathering of argentite and lead-zinc ores containing silver may release silver to the environment. ? Freshwater concentrations seldom exceed 0.04 mg/L. It is commonly used in the manufacture of silver utensils, jewelry, and solders as well as in the electroplating, photographic, and food processing industries. Other sources include wastes from silver mines, industrial electronics plants and cloud seeding.

Exposure to silver in industry is usually manifested as a condition referred to an argyria; common symptoms include a permanent blue-gray discoloration of the skin and eyes. Although excessive exposure to silver may result in kidney, liver, or spleen damage, no recognizable disturbance of health need accompany this condition. Silver is accumulated in the skin indefinitely. But skin is shed!

The maximum acceptable concentration of silver in domestic water supplies is 0.05 mg/L. This limit is used to restrict the use of silver compounds as disinfectants and is indirectly based on public health considerations. Colloial elemental silver in water is highly toxic and may result in blindness or death. Concentrations of silver in an unfiltered sample should not exceed 0.1 μg/L to protect aquatic life. source?

Sampling

Samples should be analyzed as soon as possible after collection. Silver compounds are easily adsorbed on the walls of containers.

PET plastic jars are required for aqueous samples. Add 20 drops of HNO_3 per liter of sample. Pomade jars are used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Samples are digested with an appropriate acid mixture and analyzed by atomic absorption.

Time of Analysis

Approximately 50 silver samples may be analyzed per day.

SODIUM

Sodium is the sixth most abundant element on earth, comprising about 2.6% of the earth's crust, and is found in most natural waters. Weathering of salt deposits is the principal source of sodium in the aquatic environment. Concentration levels may vary from less than 1 mg/L to several thousand mg/L in some briny groundwaters. Sodium salts are used extensively in the manufacturing of chemical products as well as in commercial water softening devices which employ the sodium ion-exchange process. It is abundant in most industrial effluents. Waters that receive surface drainage from roads during the winter season can contain high levels of sodium if sodium chloride is used to control road ice.

Soils high in sodium are generally characterized by low infiltration and permeability rates and are therefore unsuitable for farming.

Sodium is an essential element for all life forms and is generally considered non-toxic. Patients with high blood pressure are often advised to consume water with less than 50 mg/L of sodium. Waters, softened by ion exchange processes may contain sodium in excess of this amount.

Since sodium is generally considered to be non-toxic, no drinking water criteria have been established.

Sampling

Samples should be collected in PET plastic containers. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Samples are analyzed by an automated atomic absorption technique. Potassium or cesium chloride is used to minimize the partial ionization of sodium atoms in the flame.

Time of Analysis

Approximately 150 sodium analyses may be performed per day.

SOLIDS -DISSOLVED

Dissolved solids refers to the residue which is measured after evaporation of a filtered portion of sample. The test does not determine specific chemical substances but only a general class of substances which have a similar response under the test conditions. "Total solids" may be found by taking the sum of the suspended and dissolved solids.

Dissolved solids must not be added to increase ambient receiving water concentrations by more than 1/3 of the natural concentration to protect aquatic life. The added solids should not significantly alter the overall ionic balance of the receiving waters. Drinking waters should not contain more than 500 mg/L of dissolved solids.

Sampling

Samples should be collected in PET plastic containers and should be refrigerated.

Method

Dissolved solids are determined:

Gravimetrically on all industrial waste samples as well as any other type of sample where the conductivity is greater than 400 µs/cm.

From the conductivity whenever the conductivity is less than 400 µs/cm.

From the sum of constituents; Ca, Mg, Na, K, Cl, SO_4, NO_3, and alkalinity must all be requested on particular samples in order to apply this technique. This calculation is performed by the client.

Conductivity is the method of choice because it is the most precise and economical. Southern Ontario lakes and rivers free of industrial wastes have a dissolved solids content equal to their conductivity times 0.65 ± 0.10. Some caution should be exercised in interpreting the results since the calculation methods based on either the conductivity or the summation of mineral components consider only the "ionic" dissolved solids.

Time of Analysis

Two days are required to complete the gravimetric analysis. Samples are set up the first day and the weighing operation is performed the second day. Approximately 75 samples may be set up per day.

NOTE:

Total solids will be substituted for dissolved solids when filtration becomes impractical, e.g. raw sludges, digested sludges, mixed liquors, black liquor, etc.

The ignited solids test determines both the quantity of material that is lost and the ash residue when any of the total, suspended, or dissolved solids residues is ignited at 550°-600°C for one hour. The test does not determine specific substances but only a general class of substances which have a similar response under the test conditions.

The loss on ignition is due to oxidation of carbon and hydrogen, escape of water of hydration, volatilization of inorganic ammonium salts, decomposition of magnesium carbonate, and loss of any other materials which are volatile or decomposable below 550°-600°C. The loss on ignition may overestimate the organic content. The actual organic content is lower by the amount of water, inorganic salts, etc., released. In the case of clean natural waters, the loss on ignition is due almost entirely to **non-carbonaceous** material; therefore caution should be exercised in interpreting the results on such samples.

Ignited solids results are most useful in the characterization of sewage sludges where it is desirable to estimate organic composition. It is one of the major tests used in assessing the efficiency of sludge digestion.

Any inaccuracies associated with the total, suspended, and dissolved solids determinations are also an integral part of the ignited solids results. Additional errors resulting from mechanical losses due to decrepitation are also a possiblity.

Sampling

Samples should be collected in PET plastic containers and should be refrigerated. Sediment samples should be collected in pomade jars.

Method

Analysis is accomplished gravimetrically by taking the total, suspended, or dissolved solids residue and igniting at 550°-600°C for one hour. Sediment samples are ignited at 475°C for 4 hours to minimize error due to decomposition of carbonates.

Time of Analysis

Approximately 75 ignited solids determinations may be completed per day.

Suspended solids refers to the quantity of material that may be removed from a water sample by filtration using a filter of specific particle retention characteristics. The test does not determine specific chemical substances but only a general class of substances which have a similar response under the test conditions.

The measurement of suspended solids is often improperly used to assess water clarity. Clarity is a function of a water's optical properties and rarely relates to the quantity of suspended material present. Turbidity is a much better measurement of water clarity than suspended solids.

Suspended solids data are useful in characterizing domestic wastewater and in assessing the efficiency of waste treatment procedures. They are important in calculating loadings to aeration tanks, digesters, lagoons, and sedimentation tanks.

Acquiring a representative aliquot for analysis is the major source of analytical variability in the suspended solids measurement. Results on samples which contain large particles or more than 0.2% suspended material by weight are questionable because of rapid settling. Since there is considerable analytical variability in the results when the suspended solids concentration is less than 10 mg/L, samplers are urged to substitute a turbidity measurement as often as possible.

Sampling

Samples should be collected in PET plastic containers and should be refrigerated.

Method

Analysis is accomplished by filtering a known volume of sample through a pre-weighed filter paper (Whatman 934 AH : nominal particle retention is > 1.5μm). The filter is then dried overnight at 103° - 106°C. Results are reported as mg/L of suspended solids.

Time of Analysis

Two days are required to complete the analysis. Samples are filtered the first day and the weighing operation is performed the second day. Approximately 125 samples may be set up per day.

Total solids refers to the quantity of material that remains as a residue after evaporation at 103 ± 2°C. This test does not determine specific chemical substances, only a general class which have a similar response under the test conditions.

Total solids are generally estimated from the sum of the suspended and dissolved solids. Direct analyses are normally only performed on samples, such as sludges, which are very difficult to filter, or on colloidal samples which are still turbid after filtration.

Sampling

Samples should be collected in PET plastic containers and should be refrigerated.

Method

Analysis may be performed gravimetrically by evaporating an aliquot of sample overnight at 103 ± 2°C. Results may also be obtained from the sum of the suspended and dissolved solids fractions.

Time of Analysis

Two days are required to complete gravimetric analysis. Samples are set up the first day and weighed the second day. Approximately 75 samples may be set up per day.

STRONTIUM

Strontium occurs in nature as the minerals celestite and strontianite. It is not found as a native metal but it does exist in the environment as a sulphate or in combination with calcium and barium minerals. Natural compounds of strontium are relatively insoluble; therefore, its presence in domestic water supplies is generally very low. Strontium-88 is derived from weathering of strontium minerals and sedimentary rocks bearing sulphate and carbonate. The testing of nuclear bombs has caused fallout of the radioactive Strontium-90 isotope. Industrial applications of strontium and its compounds include the manufacture of metal alloys, storage batteries, paints, rubber, cathode ray tubes, tracer bullets and flares; its use, however, is not widespread. The radioactive isotope Sr-90 is one of the best Beta-emitters known, and thus provides a light-weight, long lived nuclear energy source.

The chemical behavior of strontium closely resembles that of calcium with a tendency to become concentrated in the human skeletal system. There are no known harmful effects from oral ingestion. It does not appear to be necessary for plant nutrition. There are no water quality guidelines set for non-radioactive strontium.

Sampling

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per liter of sample. Soil and sediment samples should be collected in pomade jars. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested using an appropriate acid mixture and analyzed by atomic absorption spectrophotometry.

Time of Analysis

Approximately 50 strontium analyses can be performed per day.

SULPHATE

Sulphate is an ionic component of all natural waters and is widely distributed in nature. It normally varies between 10 and 80 mg/L in surface waters, but may reach several thousand mg/L where industrial discharges are involved. These discharges include mine drainage wastes, tanneries, sulphite-pulp mills, textile plants, sulphuric acid plants and some metal working industries. Precipitation may contain considerable amounts of sulphate if industrial processes, such as the combustion of coal, emit large amounts of sulphur dioxide and sulphur trioxide to the atmosphere.

Waters high in sulphate tend to form hard scales in boilers and plumbing. Under anoxic conditions, sulphates serve as an oxygen source for bacteria which convert it to hydrogen sulphide gas. Sulphate is also believed to be responsible for "crown" corrosion in concrete sewer pipes.

The maximum sulphate concentration permissible for domestic supplies in Ontario is 250 mg/L. Concentrations above this level are non-toxic but sodium and magnesium sulphate exert a cathartic effect on the gastro-intestinal tract. Sulphate above 500 mg/L may impart a bitter taste to water as well as causing cathartic effects. Sulphate levels are generally not high enough to harm aquatic life.

Sulphate, mainly as ammonium sulphate, is a major component of air particulates. It is derived from the burning of fossil fuels.

Sampling

Surface water, sewage, industrial waste, drinking water and precipitation samples should be collected in PET plastic containers. Soil and sediment samples should be collected in pomade jars and airborne sulphate by Hi-Vol filtration.

Method

Analyses on surface, ground-water, drinking water, sewage, industrial wastes. Hi-Vol, dustfall and solid samples, after extraction, as well as precipitation, and Lo-Vol sequential filters are analyzed by ion chromatography.

Time of Analysis

Approximately 20 sulphate tests can be performed per day by dual column ion chromatography and 120 by single column ion chromatography.

SULPHATION RATE

This method is used for the determination of the sulphation rate of the atmosphere. The sulphation rate is a measure of oxidizable sulphur's reactivity, and therefore allows evaluation of the potential damaging ability of sulphur compounds. The sulphur compounds, particularly, sulphur dioxide, are converted to sulphate by a lead dioxide coated glass fibre filter. Climatic differences, interferences, differences in reactivity factors for lead dioxide batches and plate preparation techniques contribute to variable reactivities for different plates. Thus, it is difficult to compare one station to another.

Sampling

Samples are collected on a plate (lead dioxide coated glass fibre filter). These filters must be kept in a sealed container before and after use.

Method

The sulphate is separated from the other materials and analyzed using ion chromotography. Results are reported as mg SO_3/100 cm^2/day.

Sulphuric acid mist, particulate sulphate and sulphur trioxide may impinge upon the plate giving high results. Mercaptans act as negative interferents.

Time of Analysis

A plate requires approximately 30 days of exposure and a week for analysis.

NOTE: This test is now done only at the Thunder Bay laboratory.

SULPHIDE

Sulphide is formed by bacterial reduction of sulphate and organic sulphur compounds under anaerobic conditions. It is, therefore, commonly found in domestic wastewater, industrial wastewater, sludges, hypolimnions of stratified lakes and any other aquatic systems where anaerobic conditions prevail. As a result, concentrations in surface waters are negligible. Hydrogen sulphide may be found in various forms depending on the pH of the water. At pH 5, it is almost entirely found as H_2S while at pH 9, it is almost entirely HS^-. The S^{2-} anion is generally not found in natural waters. Its characteristic odour of "rotten eggs" is highly objectionable.

Sulphide is an important parameter in waste treatment monitoring. Oxidation of sulphide to sulphuric acid in concrete sewer pipes leads to "crown corrosion". Soluble sulphides in excess of 200 mg/L are toxic to bacteria and will inhibit sludge digestion. Concentrations of undissociated hydrogen sulphide should not exceed 0.002 mg/L at any time or place to protect aquatic life.

Samples containing sulphides are unstable and results must be interpreted with caution.

Sampling

Samples must be collected with a minimum amount of agitation as sulphides are highly volatile and susceptible to oxidation by dissolved oxygen. The glass container should be filled completely so that no air bubbles remain after the sample is capped. Immediate delivery to the laboratory is essential. Where possible a field analysis should be performed.

Preservation is accomplished by adding 2 mL of a 2N zinc acetate solution to the sample followed by the dropwise addition of a 5% sodium carbonate or sodium hydronide solution until precipitation is complete. The final pH must be between pH 10 an pH 11. Consultation with laboratory staff is recommended when sulphide analyses are required. Soil samples should be collected in pomade jars.

Method

Drinking and Surface Water samples are analysed by semi-automated differential pulse polarography. Samples greater than 1 ppm are analysed by Iodometric titration. The semi-quantitative lead acetate method is only used to obtain a rough estimate of the sulphide concentration. Caution must be exercised in interpreting the data as some organic sulphur compounds may release sulphide under the test conditions. Sulphide results are reported in mg/L as H_2S.

Samples may be analyzed in the field using a Hach kit. Methylene blue without gas displacement is practicable for large numbers of relatively clean samples.

Time of Analysis

Approximately 15 sulphide analyses may be completed per day.

SUSPENDED AIR PARTICULATE

Suspended particulate matter in the atmosphere is present naturally and as a result of anthropogenic activity. Natural sources include wind blown dust, volcanic ash, pollens and smoke and fly ash from forest fires. Man's activities have greatly increased levels of suspended particulates, with the highest concentrations found in industrial areas, expressways, downtown areas and in the vicinity of solid waste incinerators. Residential and rural locations report relatively low levels of air particulate matter. Siliceous material generally comprises the largest component of total suspended particulate; however, the nature of the material depends primarily upon location and it is therefore difficult to generalize.

Suspended particulate in the atmosphere can cause a reduction in visibility, alterations in sky illumination due to changes in the light scattering pattern, a decrease in the amount of solar radiation reaching the earth's surface, damage to vegetation and property and physiological reactions including allergies and irritations to the respiratory tract. Health agencies have determined that particles of less than 5 μm in diameter can enter the respiratory tract and cause health problems. The accepted standard for total suspended particulate matter in the atmosphere in Ontario is 120 $\mu g/m^3$ for 24 hours, with a geometric mean of 60 $\mu g/m^3$ for 1 year.

Sampling

Samples are collected on tared glass fibre filter paper allowing the collection of particulate having an aerodynamic diameter of less than 100 μm. Samples are collected by means of a high-flow rate blower drawing air in at a rate of 40 - 60 cfm. Exposed filters are carefully removed from the holder, folded across the middle of the 10 inch side, placed in suitably marked envelopes and mailed to the laboratory. The sample should be kept dry at all times.

Method

The high volume glass fibre filter method has been accepted by most North American agencies as the standard medium for the determination of total suspended particulate, although other filters such as Whatman No. 41, Polyflow and Microsorban may be used for special analyses.

Time of Analysis

Approximately 56 filters can be prepared, conditioned and analyzed per day.

TANNINS AND LIGNINS

Tannins and lignins are members of a large group of hydroxylated aromatic compounds (polyphenols). They are natural components of plant material and are found in surface waters as a result of normal biochemical degradation processes, or as a result of industrial discharges. They are commonly found in the effluents of the paper and tanning industries. Tannins are also applied internally to boilers in order to facilitate the handling of calcium sludge deposits and to prevent the formation of a hard scale. Tannins and lignins are undesirable because they impart objectionable taste and colors to domestic water supplies even at very low levels.

At present no separations or individual determinations of tannins and lignins are performed. Results are standardized and reported as mg/L of tannic acid. To convert this result to a lignin number, the tannic acid value should be multiplied by 2.5.

Sampling

Since tannins and lignins are subject to normal biochemical oxidation, samples should be refrigerated to retard bacterial activity. No chemical preservative should be used as this may interfere with the analysis. Immediate delivery to the laboratory is essential.

Method

Tannins and lignins in aqueous samples are determined colorimetrically after direct treatment with tungstophosphoric and molybdophosphoric acids (tyrosine reagent). As other reducing agents produce the same color, their presence may cause an over-estimation of the actual tannin and lignin content.

Time of Analysis

Approximately 40 tannin and lignin analyses may be completed in one day.

THALLIUM

Thallium occurs in the minerals crooksite, lorandite and hutchinsonite. It is also present in pyrites. Generally, the only commercial sources of thallium are flue dusts and residues from metallurgical and chemical processes such as smelting of lead and zinc ores in which thallium is a minor impurity. Thallium is generally present in only trace amounts in surface waters. The monovalent ion is the most stable form in aqueous solution.

Thallium readily alloys with a number of metals to form a number of low melting binary and higher order alloys. It has some applications in the electronics and glass industries. Thallium sulphate was an important ingredient in rodenticides and pesticides, but its toxicity to humans has curtailed this use.

The element and its compounds are toxic and should be handled carefully. Contact of the metal with skin is dangerous and may be carcinogenic to humans. Thallium has been used to treat ringworm and other skin infections but its use is limited because the margin of safety between therapeutic and toxic doses is too narrow. Thallium can inhibit photosynthetic processes and plant transpiration. In both fish and invertebrates it acts as a neuro-poison. The 96 hour LC 50 for rainbow trout is less than 10 mg/L. No drinking or livestock water guidelines have been set.

Sampling

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per liter of sample. Soil samples should be collected in pomade jars. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

The sample is digested with an appropriate acid mixture and analyzed by atomic absorption or polarograph.

Time of Analysis

Approximately 50 samples can be analyzed per day, for thallium.

TIN

Tin is found mainly in the mineral cassiterite. Most of the world's supply comes from Malaysia, Bolivia, Indonesia, Nigeria and Thailand. Tin is derived naturally from weathering processes. Tin is readily adsorbed by clay minerals so it is generally only found in trace amounts. The major sources of tin in the environment are industrial discharges.

Tin foil is used for electrical condensers, bottle cap liners and food wrappings. Tin wire is used for fuses and safety plugs and is used in the production of plate glass. Tin plating over steel is the most important use of tin. Tin cans in the food packaging are the major products. Tin alloys may be used in superconductive magnets. Tin also has a number of organic applications.

Tin and its inorganic compounds are essentially non-toxic. The amounts tolerated by the human body are far higher than would normally be ingested by eating canned foods. Many organic tin compounds are toxic, and may in addition be irritating to skin, mucous membrane and other tissues. Diethyl tin can cause severe lesions and even death. There are no guidelines for inorganic tin in drinking water. Since tin is not generally taken up by plants there are no agricultural guidelines.

Sampling

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per liter.

Method

The sample is digested with an appropriate acid mixture and analyzed by flameless atomic absorption.

Time of Analysis

Approximately 50 tin analyses can be performed per day.

TITANIUM

Titanium is found in nature as the minerals rutile, ilmenite, and sphene. It ranks eighth in the natural abundance of elements and is readily found in most soils, varying between 0.5% and 10%. The high tensile strength and corrosion resistant properties of titanium metal are valuable in the manufacture of special steel alloys which are used for structural purposes in aircraft frames and engines. Titanium metal is also used in the manufacture of electrodes, lamp filaments, and X-ray tubes. Oxides of titanium are used as a white pigment in the paint, glass, and ceramics industries. Titanium and its salts are generally extremely inert and insoluble; the notable exception is titanium tetrachloride, which is highly corrosive.

Neither plants nor animals accumulate titanium to any extent. There is no evidence that titanium plays an essential role in human or animal nutrition.

There is no known toxicity to humans, animals, or plants. Surgical appliances are generally made of titanium and various titanium derivatives have been used in the treatment of skin disorders. No limits have been specified for the maximum acceptable concentration of titanium in domestic water supplies.

Sampling

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per liter of sample. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested with an appropriate acid mixture and analyzed by atomic absorption or Inductively Coupled Plasma (ICP) - atomic emission. Soils are analyzed by X-Ray Fluorescence.

Time of Analysis

Approximately 50 titanium analyses can be performed per day.

AQUATIC TOXICITY

Aquatic Toxicity is the property of a substance, effluent, sample, etc. to cause stress or unacceptable effects on aquatic life. The most commonly used laboratory measure of aquatic toxicity is the 96 hour static LC50 bioassay using rainbow trout. It results in an estimate of the concentration required to kill 50% of the trout by the end of a four day exposure period. This test is performed by the Aquatic Toxicity Unit, Water Resources Branch, Rexdale laboratories (contact Gary Westlake 416-235-5797). There are a number of other bioassessment techniques performed at the Rexdale laboratories. For genetic/mutagenic assessments contact David Rokosh 416-235-5787. For field assessments contact Wolfgang Scheider 416-323-4925.

Bioassays are direct measures of the biological effects of toxic contaminants and complex mixtures under controlled laboratory conditions. It is not necessary to know causitive agents before performing these tests and they do not usually establish the substances involved. Used in combination with chemical analyses, these can be effective assessment procedures.

Sampling

Aqueous samples should be collected in plastic pails preferably with bag liners (these can be obtained from the Rexdale laboratory). The sample should fill the container with no head space and should be maintained as cool as possible but not frozen. A total volume of 80 litres is normally required. Although, special arrangements can be made for smaller volumes. The aquatic toxicity lab should be warned when each sample is coming due to schedules and should arrive in the lab within 2 days of sampling, preferably as soon as feasible. The sample normally cannot be used if it is older than 5 days.

Method

A concentration series including a control in 20 litre pails is prepared and maintained at 15°C during the test. 10 trout are placed in each and observed over four days. A statistical assessment of these observations gives the LC50 values and confidence intervals. Since this data cannot be made to fit the storage formats available on LIS or SIS, a report is sent directly back to the clients. In this report, where feasible, interpretation is given on potential causes for toxicity in the sample.

Time of Analysis

A maximum of seven bioassays can be performed at one time. The annual capacity of the aquatic toxicity unit is approximately 200. An additional 200/year can be done under contract with Lakehead University.

TRACER DYES

Sodium fluorescein is used as a tracer dye to follow ground water movement. It may also be used to monitor effluent from septic tank systems, especially where there is suspected contamination in drinking water. Concentrations can be reported but is of little significance. Relative concentrations in a set of samples are more useful to the sampler. The test is generally only used to detect the presence of the dye. Rhodamine dye can also be determined by special request.

Sampling

Samples should be collected in glass bottles and kept in a cool, dark place to minimize photodegradation activity and oxidation.

Method

Analysis is performed using a fluorospectrophotometric technique.

Time of Analysis

Approximately 20 samples may be analyzed per day.

TURBIDITY

Materials present in water absorb, scatter and reflect light. Dissolved materials absorb light but a substantial reduction in light transmission is due to the presence of suspended particles. Turbidity is a measure of the optical properties of a sample which cause light to be scattered rather than transmitted in straight lines through the sample. In water samples, turbidity is a result of suspended clay, silt, finely divided organic and inorganic matter, plankton and other microscopic organisms. While shoreline erosion, resuspension of sediment material and algal blooms decrease light transmission naturally, human activities can also have an effect.

Turbidity may not be correlated with the suspended solids test because the size, shape, refractive index and hence, the light scattering effect of the suspended matter have little relationship to their concentration and specific gravity.

Water clarity is one of the main criteria used by the public to judge water quality for drinking and recreational use.

Care must be used in interpreting turbidity results since these are defined by the instrument, the standards and the optical phenomena being measured. Standards other than Formazin are exceedingly difficult to reproduce as their optical properties depend on the sizes, shapes and refractive indices of the component particles in suspension.

The turbidity of the water can have a great effect on the types and quantities of algae that grow in it by altering the amount of light available for photosynthesis. Suspended matter should not be added to surface waters in concentrations that will change the natural Secchi disc reading by more than 10 percent.

Sampling

Samples should be collected in PET plastic containers. Samples should be kept in the dark and delivered to the laboratory within 24 hours. Inclusion of rapidly-settling particles in a sample makes a representative reading impossible (the instrument cannot provide a stable output).

Method

The instrument is standarized with a sealed standard which is prepared commercially from latex polymers of known size and rated in Formazin Turbidity Units. Samples are placed in the turbidimeter, and results in FTU are read directly from the digital output. Turbidity measurement are based on light scattering at 90 plus or minus 30 degrees of rotation. The instrument compensates for sample color.

Time of Analysis

Approximately 150 turbidity determinations can be performed per day.

URANIUM

Uranium is found in nature as the minerals carnotite and uraninite as well as in igneous type rocks. It is well known for its radioactive properties since its major industrial application is the manufacture of nuclear fuel. Natural uranium, however, is present as the isotope U-238, which is not strongly radioactive. Uranium in natural waters is derived from weathering of uranium minerals and acid igneous rocks and from discharges from nuclear fuel processing plants and from photography and glazing industries.

In natural waters uranium is usually hexavalent and commonly present as the uranyl ion UO_2++. Uranyl salts are very soluble and produce an objectionable taste and color when they exceed a concentration of 5 mg/L as uranium. At considerably lower levels, ingestion of water containing uranyl ions may result in possible kidney damage. Limits on the maximum concentration of uranium in domestic water supplies, when specified, are generally based on chemical properties of the uranyl ion rather than on radioactivity. At present, an interim limit of 0.02 mg/h is recommended for drinking water.

Sampling

Aqueous samples are collected in PET plastic containers and preserved with 20 drops of HNO_3 per liter of sample. Soil and sediment samples should be collected in pomade jars. Vegetation samples are collected in perforated plastic bags and must be refrigerated.

Method

Water samples are analyzed by Inductively Coupled Plasma Mass Spectrometry. Sewage and industrial waste samples are analyzed by a reflective fluorescence technique.

Time of Analysis

Approximately 50 uranium analyses can be performed per day.

VANADIUM

Vanadium occurs in nature as the minerals vanadinate, patronite, mottramite, and carnotite, and is generally found in the presence of selenium. Metallic vanadium does not occur in nature. Trivalent vanadium salts tend to be insoluble but tetra- and pentavalent salts tend to be soluble. Seepage from carbonaceous deposits may be a major source of vanadium. Other sources include weathering and industrial discharges. Vanadium is used in the steel industry where it is known to increase the general toughness and tensile strength of certain alloys, but it is also used in the manufacture of dyes, paints, inks and ceramics. Vanadium has been found in all fuel and motor oils and has been used for oil spill identification by means of the nickel-vanadium ratio. Vanadium in surface waters is generally found at levels below 0.05 mg/L.

The usefulness of vanadium in animal and plant metabolism has not been convincingly demonstrated and its behaviour in biological systems is not well understood. Some experimental evidence suggests that the presence of vanadium in trace quantities may be beneficial in the prevention of heart disease.

Several compounds of vanadium are known to be toxic in high concentration. Exposure may produce nausea, diarrhea, abdominal pain, nervous trembling of the hands, and greenish-black discoloration of the tongue. No limits have been specified for the maximum concentration of vanadium in domestic water supplies because the element is rarely encountered. Levels in livestock drinking waters should not exceed 0.1 mg/L.

Sampling

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per liter of sample. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested using the appropriate acid mixture and analyzed. Hi-Vol samples are analyzed by Inductively Coupled Plasma (ICP) atomic emission. All other samples are analyzed by atomic absorption.

Time of Analysis

Approximately 50 vanadium analyses may be performed per day.

ZINC

Zinc is commonly found in nature as zinc sulphide in the mineral sphalerite and as zinc carbonate in the mineral smithsonite. It is widely used in the manufacture of metal alloys such as brass, batteries, dyestuffs, paints, ointments and rubber, as well as in electroplating. Zinc oxide is used in medical ointments and as a pigment. Zinc carbonate and sulphide are used as white pigments in paints and enamels. The sulphide is also used as a phosphor for television screens and fluorescent coatings. Zinc most commonly enters a water supply via the deterioration of galvanized iron, the dezincification of brass, direct industrial discharges, and drainage from mine wastes. It is usually found in concentrations less than 0.05 mg/L in most natural waters but acidic waters may hold up to 50 mg/L.

Zinc is essential to plant and animal life. It is a component of several metalloenzymes and proteins. Deficiencies result in growth retardation, skin lesions, bone weaknesses, poor healing of wounds, impaired reproductive processes and reduced learning capacity.

Plants growing in a zinc deficient soil are severely stunted. In animals, zinc is an important constituent of a number of enzymes, notably carbonic anhydrase which is vital for normal respiration. Zinc and its compounds are relatively non-toxic when taken orally. Even at high concentrations, nausea and diarrhea are the only adverse effects likely to be encountered. The limiting factors which determine the acceptable maximum concentration in a water supply are taste and appearance. Zinc, in excess of 5 mg/L, imparts a bitter astringent taste when the water is boiled and may give a milky appearance in alkaline waters. The maximum acceptable limit for domestic water supplies in Ontario is therefore 5 mg/L. Concentrations of zinc in an unfiltered sample should not exceed 0.030 mg/L to protect aquatic life. Zinc toxicity increases with increasing temperature and decreasing dissolved oxygen. The presence of copper and cadmium enhance the sublethal toxicity of zinc.

Sampling

PET plastic containers are required for aqueous samples. Add 20 drops of HNO_3 per liter of sample. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Samples are digested with the appropriate acid mixture and analyzed. Sewage, sludge and Hi-Vol samples are analyzed by Inductively Coupled Plasma (ICP) atomic emission. All other samples are analyzed by atomic absorption spectrophotometry.

Time of Analysis

Approximately 50 zinc analyses may be performed per day.

SPECIALIZED CAPABILITIES

In addition to the tests outlined in this manual there is available a high degree of specialized expertise and equipment for conducting project work and special studies. Such specialized work often requires a great deal of planning and is time consuming. It cannot be considered part of the routine work and may require specific consultations and funding.

Some specialized capabilities are listed below.

- Microscopic identification of materials (particulates, algae, fungi, pollen, etc.)
- Metal scanning in various matrices by powder spectrograph and Inductively Coupled Plasma (ICP) atomic emission.
- Electron microscope and electron probe analysis of particulate matter in water and air, of soil, sediment and special materials including bacterial cells. All metals can be scanned qualitatively and semi-quantitatively.
- Polarographic procedures, such as differential pulse anodic stripping voltametry (ultra trace concentrations of certain metals, speciation of ions and complexation.
- X-ray diffraction identification of crystalline compounds.
- Gas chromatography/mass spectrometer analysis of organic compounds.
- Ion chromatograph scanning for inorganic anions in water, precipitation and air samples. This is a quantitative scan.
- Long term oxygen uptake measurements on wastes and sediments using oxygen producing probes.
- Analysis of component parts from a sampling train (probe filters, impinger fractions) inserted in a stack. These are usually analyzed for routine metals and anions.
- Biomass determinations (direct count, ATP and Coulter counter).
- Metabolic activity (respirometer, continuous culture).
- Microbiological transformation of environmental contaminants (batch culture, continuous culture, respirometer studies).
- Detection of genotoxic activity in pure subtances and environmental samples. Methods now available include the Ames and Rosencrantz tests.
- Detection and isolation of viruses from natural waters, sediments and sewage samples.
- Determination of plasticity, permeability and cation exchange capacity for soils and sediments; settleability of dredged spoil.
- Analysis of extractions and leachates of soil, sediment and solid industrial waste samples.
- Waste characterization for Reg. 309 including slump test, corrosivity test, flash point, etc.
- Organic characterization for complex samples.

TABLE I - ENQUIRES - SAMPLING AND TEST PROCEDURES, SAMPLE STATUS

Enquiries regarding sampling and test procedures and the status of outstanding samples should be directed to the appropriate individuals listed below.

All samples received by the Laboratory Branch are assigned alpha-numeric codes according to sample type. As the analyses are completed, the results are entered into the computer (LIS system). When data for all tests have been entered, final reports are checked by senior staff and mailed. All original submission sheets are retained in the sample reception files for 18 months, after which they are destroyed. Laboratory staff are prepared to answer questions regarding the receipt and progress of samples but require the following information:

a) Submission number and date of submission
b) Name of sampling area as given on submission
c) Field sample number and sample description
d) Type of sample (e.g. water, river, sewage, industrial wastes, Great Lakes, algae, etc.)

NOTE: The submission number is the most important piece of information in tracking work through the Laboratory Information System. Please retain this record in a safe place and use it when requesting information as to the progress of your analytical work.

TABLE I - ENQUIRES - SAMPLING AND TEST PROCEDURES, SAMPLE STATUS (cont'd)

WATER QUALITY SECTION

Telephone - (416) 235-5870

Manager	Larry Vlassoff	(416) 235-5870
*ERTF Representative	Vera Turner	(416) 235-5873

The Section is responsible for the analysis of all major ions, nutrients and physical properties in water (including precipitation, surface water, groundwater, sewage and industrial wastes). The section is also responsible for environmental microbiology tests.

General inquiries regarding results should be directed to the following staff based on sample classification.

Supervisors (Contact for General Discussion, Planning, Problem Samples, etc.)

	Contact Staff	**Telephone**
Provincial Water Quality Monitoring	Walter Wright	(416) 235-5879
Surface Water	Joan Crowther	(416) 235-5868
Acid Rain and Air, Dorset Lab	Frank Tomassini	(416) 235-5869
Wastes and MISA	Peter Campbell	(416) 235-5872
Drinking Water	Mike Rawlings	(416) 235-5880
Drinking Water (Microbiology)	Jim Clark	(416) 235-5883
Lakes, Rivers & Wastes, Nuisance Organisms (Microbilogy)	Michael Young	(416) 235-5866
Court Cases	Vera Turner	(416) 235-5873

*ERTF = Emergency Response Task Force

TABLE I - ENQUIRES - SAMPLING AND TEST PROCEDURES, SAMPLE STATUS (cont'd)

WATER QUALITY SECTION
Telephone - (416) 235-5870

Classification Name	LIS Sample Class	Analytical Consulting Staff	
Chemical:			
-Great Lakes	G	P. Millar/ J. Crowther	(416) 235-5882
-Public Health Inspection	H	J. McBride/J. Crowther	(416) 235-5875
-River (surface water)	R	W. Wright/J. Crowther	(416) 235 5879
-Sewages (UTOP, M & P)	S	B. Cheung/V. Turner	(416) 235-5874
-Industrial Waste	I	V. Turner/B. Cheung	(416) 235-5873
-Precipitation	P	B. Wright/J. McBride	(416) 235-5870
-Water (Ground/Domestic)	W	J. McBride/M. Rawlings	(416) 235-5875
-APIOS Air Filters	AS	B. Wright/J. McBride	(416) 235-5870
-Leachates	LW	S. Tracy/V. Turner	(416) 235-5877
-Soil Leach - ARB (APIOS)	SX	B. Wright/J. Crowther	(416) 235-5870
-High Priority/Court Cases	CC, ERTF	V. Turner/B. Cheung	(416) 235-5873
-Dorset Precipitation, Rivers & Lakes	DP, DR	C. Chun/P. Sutey	(705) 766-2412
-Dorset Soil Leach, Stemflow	DX, DS	A. Neary	(705) 766-2412
-Dorset Terrestrial Effects	TE	A. Neary/S. Barnes	(705) 766-2412
Microbiology:			
-Drinking Water	MW	S. Debrecini/J. Clark	(416) 235-5884
-Lakes, Rivers & Wastes	MS	S. Janhurst/M. Young	(416) 235-5888
-Nuisance Organisms, Taxonomy	MT	G. Horsnell/M. Young	(416) 235-5886

TABLE I - ENQUIRES - SAMPLING AND TEST PROCEDURES, SAMPLE STATUS (Cont'd)

INORGANIC TRACE CONTAMINANTS

Telephone - (416) 235-5845

Manager	Barry Loescher	(416) 235-5848
ERTF Representative	Liz Pastorek	(416) 235-5855

The ITC Section provides analytical services which include the determination of elemental and mineral constituents in a variety of sample types such as soils, vegetation, air filters, fish, etc.

General inquiries regarding results should be directed to the following staff based on sample classification.

Classification Name	LIS Sample Class	Contact Staff	
Biomaterials	B	R. Sadana	(416) 235-5845
Fish	F	R. Sadana	(416) 235-5845
Metals in Water	M	P. Vijan	(416) 235-5858
Fluoride Candles	FC	Brian Foster/David Sturgis	(416) 235-5853/235-5850
Hi-Vol Filters	HV	"	"
Anderson Filters	AF	"	"
Dustfalls	DF	"	"
Hi-Vol Particulates	HP	"	"
Dichotomous Metals	DC	"	"
Industrial Wastes	IW	Jim Pimenta/George Wood	(416) 235-5854/-5845
Landfill Leachates	LF	" "	"
Sediments/Soil	SE, SO	Liz Pastorek	(416) 235-5855
Sewage Sludge	SW	Jim Pimenta	(416) 235-5854
Precipitation, Stemflow	AI, PI, SM	P. Vijan	(416) 235-5858
Terrestrial Effects	TE, TX	Liz Pastorek	(416) 235-5855
Vegetation	V	" "	(416) 235-5855
Court Cases	CC	Rusty Moody	(416) 235-5863
Particulate Identification/Complaint	E	" "	
Electron Microscopy, Legal	E, RC, CC	" "	(416) 235-5863

TRACE ORGANICS SECTION

Telephone - (416) 235-5758

Manager (Acting)	George Crawford	(416) 235-5757
ERTF Representative	Joe Osborne	(416) 235-5759
MISA Coordinator	Wendy Moss	(416) 235-5761
Contaminants Research Scientist	Dale Sutherland	(416) 235-5758

The Section is divided into four main functional units:

The current analytical capabilities of each are:

1. Biota and Solids:

Currently the unit has capabilities for a broad range of extractable pesticides, herbicides, chlorophenols, PCBs, chlorinated benzenes, toluenes and higher molecular weight chlorinated industrial organics, and a limited range of other industrials such as phosphate ester plasticizers for hydraulic fluids.

2. Air Analysis:

Currently the Air Analysis unit has capabilities for sampling and analysis for a range of pesticides, PCBs, and chlorinated industrial chemicals in the vapor phase. Capacity (4-5 samples per day) is available for analysis of organic vapors (both chlorinated and non-chlorinated compounds) in ambient air by absorbent trap-thermal desorption.

This method is being extended to analyse chlorinated species at much higher sensitivities. The unit has the capability for limited non-automated analysis of gas samples for gaseous hydrocarbons, sulphur gases, and chlorinated hydrocarbons.

3. Hazardous Waste Unit/Mobile Operations:

This unit has three functions: (a) co-ordination of all LSB regional mobile landfill investigations and/or on site monitoring studies, and determining related organic constituents; (b) the analysis of solids and hazardous/high concentration liquid wastes for toxic organics; (c) characterization of solid and liquid wastes for investigations and litigation.

TABLE I - ENQUIRES - SAMPLING AND TEST PROCEDURES, SAMPLE STATUS (Cont'd)

TRACE ORGANICS SECTION

Telephone - (416) 235-5758

4. Wastewater Unit:

This unit has two main functions: (a) to provide analytical services for the measurement of trace organics in municipal and industrial effluents, leachates, etc.; (b) to provide specialized advanced analytical instrumentation services to the rest of the section.

Based on the above criteria, general inquiries regarding trace organic results should be directed to the following staff based on sample classification.

Classification Name	LIS Sample Class	Contact Staff
		(Phone (416) 235-5758)
Sediments/Soils	PS	Dr. Ed Adamek
Biomaterials (not fish)	PB	"
Biomaterials (fish)	PF	"
Vegetation	PV	"
Air, Precipitation	PA, WX	Dr. Brian Foster
Landfill (any)	CH, PL	Bob Guttridge
Water (any)	PW	Yvonne Jones
Trade Wastes	PT	Pat Baulu
Mobile Operations	PM	Joe Osborne
Characterization	SP	George Wyhovsky

TABLE I - ENQUIRES - SAMPLING AND TEST PROCEDURES, SAMPLE STATUS (Cont'd)

DRINKING WATER ORGANICS SECTION

Telephone - (416) 235-5900

Manager	Helle Tosine	(416) 235-5906
ERTF Representatives	David Hall	(416) 235-5910
	Vince Taguchi	(416) 235-5902

General inquiries, should be directed to the following staff based on parameter:

Parameter	LIS Sample Class	Matrix	Scan	Contact Staff
Polychlorinated dibenzodioxin and	PX	fish		R.E. Clement ((416) 235-5896)
Polychlorinated dibenzofurans	DN	incinerator emissions water, biota soils/sediments special projects		
Trihalomethanes	OP	drinking, ground and surface waters	OCOHVI	O.W. Berg ((416) 235-5907)
Volatile/purgeable organics			OPOPUP (OPOPRO) - on clean samples only	
Base/neutral/acid EPA			OPOBNO " - on clean samples only	
Extractables			OPOAXO " - on clean samples only	
Extractables MSD			OPODMS - on clean samples only	
PCB's	OW	drinking, ground and surface waters	OWOC	D. Hall ((416) 235-5910)
Chlorinated aromatics			"	
Organochlorine pesticides			"	
Chlorinated phenols			OWCP	
Phenoxy acid herbicides			"	
Speciated Phenols			OAPHNX	David Hall ((416) 235-5910)
Special PAH's			OAPAHX	
Organophosphate pesticides		sediments/soils water/effluents. Plant tissues only on consultation with lab.	PEOP	
Carbamates		"	PECAR	
Triazines		"	OWTRI	
Phenylureas		"	PEURH	
Parameter Class				
Mass Spectrometry, Extractables	MY	"	PBEXT	Vince Taguchi ((416) 235-5902)
GC/MS Volatiles			PBVOL	

(no page 131)

TABLE I - ENQUIRES - SAMPLING AND TEST PROCEDURES, SAMPLE STATUS (Cont'd)

AQUATIC BIOLOGY SECTION (WATER RESOURCES BRANCH)

	LIS Sample Class	Contact Staff	
Aquatic Plant Unit: Phytoplankton, Zooplankton, Periphyton	GH	Gord Hopkins Ken Nicholls	(416) 235-5811 (416) 235-5810
Aquatic Toxicity Unit		Gary Westlake	(416) 235-5797
Bioassessment Unit		Wolfgang Scheider	(416) 235-4925
Biohazard Unit		Dave Rokosh	(416) 235-5787

LABORATORY COMPUTER SYSTEMS - QA/QC SECTION

Manager		Paul Roberts	(416) 235-5841
LIS/SIS Coordinator		" "	"
Quality Assurance/Quality Control		Don King	(416) 235-5838

SHIPPING AND RECEIVING

Central Stores		Ron Harrison	(416) 235-5741
Sample Bottle Supply		Julian King	(416) 235-5740

LONDON REGIONAL LABORATORY

Chemistry		Walter Cook	(519) 681-3600
Microbiology		Gary Palmateer	"

THUNDER BAY REGIONAL LABORATORY

Manager		Jim Stasiuk	(807) 475-1275
Chemistry		Patrick Leung	"
Microbiology		Stuart Irwin	"

KINGSTON REGIONAL LABORATORY

Chief Laboratory Services		Stan MacBeth	(613) 549-4000
Chemistry		Dave Ferguson	"
Microbiology		Art Ley	"

TABLE 2 - SAMPLE CONTAINER REQUISITION

SAMPLING SUPPLIES - CENTRAL STORES (416) 235-5746

*** To be replaced by polyethylene terephthalate (PET) jars (500 mL)**

**** To be replaced by 250 mL WM Glass**

WM = Wide Mouth

Type of sample	Container	Pack contents	Pack No.	Special Features analysis
		CHEMICAL SAMPLING CONTAINERS		
Water, liquid wastes - general purpose	1 L Glass*	6 bottles (3 x 2)	Pack #3*	foil liner in cap
Water; Public Health domestic inspections	200 mL Plastic	10 bottles in sleeves	Health Pack	Includes sleeves, mailing labels, submission form
Sludges, sediments -general purpose including metals	1 L WM Glass* 500 mL WM Glass	6 jars (3 x 2) 12 jars (3 x 4)	Pack #4* Pack #5	
Low level nutrients	250 mL WM Polystyrene	12 jars (3 x 4)	Pack #12	freezable
PCB, pesticides, organics	1 L Brown Glass 500 mL WM Glass**	6 bottles (3 x 2) 12 jars (3 x 4)	Pack #3P Pack #5P**	foil liner, container washed with solvents
PCB's in oil	30 mL glass	24 jars	Pack #21	
Phenols	250 mL Glass Green Label	4 bottles 48 bottles	Pack 7P Pack #8P	white cap: dull foil or Teflon liner; pres. added
General chemical tests	250 mL glass	4 bottles 48 bottles	Pack #7TC Pack #8TC	black label, unsterilized recycled
Chlorophenols/ phenoxy acids	1 L Brown Glass	6 bottles (3 x 2)	Special #3	solvent rinsed, baked; Teflon lined cap
Purgeables & Trihalomethanes	250 ml Glass	4 bottles	Pack 7TC	black label, foil lined cap; **NO PRESERVATIVE!**
Metals, precipitation and low level nutrients	500 mL WM Polystyrene*	12 jars (4 x 3)	Pack #20*	use once only

TABLE 2 - SAMPLE CONTAINER REQUISITION

SAMPLING SUPPLIES - CENTRAL STORES (416) 235-5746

*** To be replaced by polyethylene terephthalate (PET) jars (500 mL)**

WM = Wide Mouth

Type of sample	Container	Pack contents	Pack No.	Special Features analysis
		MICROBIOLOGICAL SAMPLING CONTAINERS		
Unchlorinated water	250 mL Glass Red Label	4 bottles	Pack #7T or #7TF	Pre-sterilized, thiosulphate added (F = foampack)
Rivers, lakes, wells; Chlorinated water	" "	48 bottles (4x12)	Pack #8T or #8TF	
		AQUATIC PLANT SAMPLES		
Lake, river, raw water, wells	1 L Glass	6 bottles (3 x 2)	Pack #3S	Express
Shoreline accumulations and scums	500 mL WM Glass or *	12 jars (3 x 4)	Pack #5 or *	Use rubber cap liner (not foil if preservative added)

TABLE 3 - SPECIFIC PARAMETER INFORMATION

A number of the determinations listed in Table 3 may be applied to several sample matrices. The information in the table is for aqueous samples unless otherwise stated. Other eligible matrices are indicated by initial in parentheses in the comments column, and sampling information is available from the appropriate laboratory section. Ambient air samples are collected in aluminized plastic bags, 5 to 22 L.
H = Hi-Vol filters; V = Vegetation; S = Sediments & Soils; A = Ambient Air; B = Biomaterials, Fish; D = Dustfall; P = Aquatic Plants

Parameter	Container	Preservation Technique	Minimum Volume Required	Comment or Common Name
MAJOR IONS				
Alkalinity	Glass or Plastic	None	75 mL	
Calcium	" "	"	75 mL	
Chloride	" "	"	50 mL	
Conductivity	" "	"	75 mL	Specific Conductance
Hardness	" "	"	75 mL	
Magnesium	" "	"	75 mL	
Potassium	" "	"	40 mL	
Silicates - Reactive	Plastic only	"	50 mL	Silica
Sodium	Glass or Plastic	"	40 mL	
Sulphate	" "	"	50 mL	
NUTRIENTS				
Ammonia Nitrogen (Filtered)	Glass or Plastic (not linear polyethylene)	Freeze or Refrigerate	75 mL	Free Ammonia
Nitrate Nitrogen			"	
Nitrite Nitrogen	"	"	"	
Nitrogen - Total Kjeldahl	"	"	"	
Phosphorus - Total	"	"	"	
Phosphorus - Filtered Reactive	"	"	"	Soluble Phosphorus, Orthophosphate

TABLE 3 - SPECIFIC PARAMETER INFORMATION (Cont'd)

Parameter	Container	Preservation Technique	Minimum Volume Required	Comment or Common Name
METALS				
Aluminum	Plastic or Glass(2)	HNO_3 to pH of 2 (approximately 20 drops per liter)(1)	100 mL; 900 mL required for preconcentration ultra-trace analysis)	(H, V, S, D, B)(4)
Antimony	"			
Barium	"			
Beryllium	"	"	"	"
Cadmium	"	"	"	"
Chromium	"	"	"	"
Cobalt	"	"	"	"
Copper	"	"	"	"
Iron	"	"	"	"
Lead	"	"	"	"
Lithium	"	"	"	"
Manganese	"	"	"	"
Molybdenum	"	"	"	"
Nickel	"	"	"	"
Silver	"	"	"	"
Strontium	"	"	"	"
Tellurium	"	"	"	"
Thallium	"	"	"	"
Titanium	"	"	"	"
Uranium	"	"	"	"
Vanadium	"	"	"	"
Zinc	"	"	"	"
Arsenic	"	None	50 mL	(V, S, D, B)
Boron	Plastic only	HNO_3 as above	100 mL	(V, S, D, B)
Chromium - hexavalent	Glass only	None	100 mL	(V, S, D)
Mercury	Glass only	$HNO_3 + K_2Cr_2O_7$(3)	200 mL	(V, S, B)
Selenium	Plastic or Glass[2]	HNO_3 as above	100 mL	(H, V, S, D, B)

[1] Nitric acid preservative should be added AFTER the sample is placed in the bottle. If the sample contains visible suspended solids or where a hazardous chemical reaction between the sample and the acid may occur, submit the sample unpreserved.

[2] Acid washed polyethylene or new (unwashed) polystyrene containers are required for ultra-trace analysis; glass bottles must have non-metallic cap liners.

[3] A special milk dilution bottle (stores #8c) is used for Hg samples. Add 1-2 mL HNO_3 per 250 mL, followed by at least 10 drops of $K_2Cr_2O_7$. The $K_2Cr_2O_7$ solution should produce a definite yellow colour. Omit preservation where dangerous reactions may occur, or where the sample is heavily contaminated with organic material. If in doubt contact Darryl Russell (248-3023).

[4] Most of the metals listed can be determined also on Hi-Vol filters (H), vegetation (V), sediment/soils (S), and dustfall (D); some of the metals listed can be determined on biomaterials (B). For specific metals, contact appropriate I.T.C. staff (Table I).

TABLE 3 - SPECIFIC PARAMETER INFORMATION (Cont'd)

Parameter	Container	Preservation Technique	Minimum Volume Required	Comment or Common Name
ORGANIC				
Acid extractables	Glass (6)	Refrigerate	1 L	(7)
Base/neutral extractables	Glass (6)	Refrigerate	1 L	(7)
Benzene Soluble Organics				(D, H, S, B, A)
Biochemical Oxygen Demand	Glass	Refrigerate	500 mL	BOD$_5$ - Please Mark Lid With Green Tape
Carbon - Free (Elemental)				(H, S)
Carbon Dioxide	Special (5)	Refrigerate	-	Free CO_2
Carbon - Dissolved Inorganic	Glass or Plastic	"	50 mL	
Carbon - Dissolved Organic	Glass or Plastic	"	"	
Carbon - Inorganic	Glass or Plastic	"	"	(H, S)
Carbon - Total	Glass or Plastic	"	"	(H, S)
Chemical Oxygen Demand	Glass	Refrigerate	25 mL	C.O.D.
Dioxin	-Consult with Drinking Water Organics (DWO) Section -R. Clement/B. Bobbie (416) 235-5900			
Foams		Glass	Refrigerate	-Consult Trace Organics (TO) Sectio
Freons		Glass	Consult with TO Section	(A)
G.C./M.S. Characterization	Brown Glass, foil liner (6)	Refrigerate	2 L	Consult Mass Spectrometry Unit
Hexachlorobenzene (OWOC Scan)	Glass (6)	Refrigerate	(6)	HCB
Hydrocarbon Gases	Glass	Refrigerate	1 L	(A)
Mercaptans - see Volatile Sulfurous Organics				
Methane - see Hydrocarbon gases				

TABLE 3 - SPECIFIC PARAMETER INFORMATION (Cont'd)

Parameter	Container	Preservation Technique	Minimum Volume Required	Comment or Common Name
ORGANIC				
Petroleum Hydrocarbons(Gasoline)	Glass	"	1 L	
Phenolics - Reactive	Glass	$CuSO_4$ - H_3PO_4	250 mL	A special bottle containing preservative is available.
Polybrominated Biphenyls	Glass (6)	Refrigerate	(6)	PBB
Polychlorinated Biphenyls	Glass (6)	Refrigerate	(6)	PCB (for drinking, surface and ground water, use (OWOC) scan)
Polynuclear Aromatic Hydrocarbons	Glass	"	1 L	PAH
Purgeable Organics	Glass	"	250 mL	No preservatives
Resins and Fatty Acids	Glass	Adjust to pH 3 with HCl; refrigerate	1 L	
Solvent Extractables	Glass	Refrigerate	1 L	Ether Solubles
Tannins and Lignins	Glass	"	200 mL	
Tracer Dyes	Consult with Trace Organics Section			
Trihalomethanes	Glass	"	250 mL	No preservatives
Vinyl Chloride	Glass	Refrigerate	1 L	(A)
Volatile Sulfurous Organics	Glass	"	1 L	(A)

5. CO_2 samples are to be carefully transferred from the sampling device into the bottom of a leak-proof glass stoppered container so as to prevent splashing. After copious overflow the bottle must be stoppered excluding air and rushed to a laboratory.
6. Special solvent washed and baked containers are supplied. Fill container to mark, approx. 900 mL.
7. The same sample may be used for both acid and base neutral extractables.

TABLE 3 - SPECIFIC PARAMETER INFORMATION (Cont'd)

Parameter	Container	Preservation Technique	Minimum Volume Required	Comment or Common Name
OTHER				
Acidity	Glass or Plastic	Refrigerate	50 mL	Precip. or surface water samples
Asbestos	Plastic	Refrigerate Analyze within 48 hours.	1 L	(H, special filter)
Chlorine - Total Residual,	Glass	(9)	500 mL	(H, V)
Free Available, Monochloramine,	"	(9)	500 mL	
Dichloramine	"	(9)	500 mL	
Chlorophyll	Field filtration preferred	(8)	500 mL	
Colour - Apparent	Glass or Plastic	Refrigerate	75 mL	Hazen Colour Units
Colour Dilution	Glass or Plastic	Refrigerate	50 mL	
Cyanide	Glass or Plastic	NaOH to pH 11+	500 mL	
Dustfall	See text			
Fluoridation Rate	See text			
Fluoride	Glass or Plastic	Refrigerate	50 mL	(H, V, B by ISE (11))
Loss on Ignition	Glass or Plastic	Refrigerate	500 mL	(D, H, S, V);
Oxygen - Dissolved	Glass	(9)	600 mL	Use BOD bottles
Particle Size Analysis	Sediment samples only; Contact ITC			
Particle Size by Microscopy	Non-aqueous samples only; Contact EM Unit			
Particulate Identification (Complaint Samples)	D,V,P; ITC, or Aquatic Biology Unit, Water Resources Branch.			

(8) In the field, filter up to 1000 mL sample (minumum volume required 500 mL) through one nylon membrane filter (1.2 um pore size, 47 mm dia: Central Stores Cat. No. F036-1). Fold the filter in half avoiding exposure of the suspended matter, and place it on a filter pad (Cat. No. F066-1) in a plastic Petri dish equipped with a cover (Cat. No. D069-1). Record the volume (to the nearest 10 mL) of sample, which is filtered, on the Petri dish. If field filtration is not feasible, submit one litre of sample in a glass container. **For either dishes or bottles, protect samples from the light, refrigerate, and ship as soon as possible.**

NOTE: For chlorophyll analyses, the laboratory welcomes two filters or two bottles of sample. These replicates are needed to obtain duplicate data for our quality control program.

TABLE 3 - SPECIFIC PARAMETER INFORMATION (Cont'd)

Parameter	Container	Preservation Technique	Minimum Volume Required	Comment or Common Name
OTHER				
pH	Glass or Plastic	Refrigerate	50 mL	
Settleability	Glass	"	900 mL	
Sieve Analysis	-	-	-	Non-aqueous samples only
Silicon	-	-	-	"
Solids - Filtered	Glass	Refrigerate	75 mL	
Solids - Ignited	"	"	600 mL	See also Loss on Ignition
Solids - Suspended	"	"	"	
Solids - Total	"	"	75 mL	
Sulfation Rate	-	-	-	See text - Section III.B.II.5
Sulfide	Glass or Plastic	Zn acetate + $\mathrm{Na2CO_3}$ (10)	900 mL	Consult Inorganic Trace Contaminants Section prior to sampling
Sulfite	"	Refrigerate	1 L	
Sulfur - Total	-	-	-	Non-aqueous samples only
Suspended Air Particulates - Total	-	-	-	TSP
Thiocyanate	Glass or Plastic	NaOH to pH 11+	"	
Turbidity	"	Keep in darkness	50 mL	

(9) Due to the perishable nature of the measured constituents, analysis should ideally be performed on-site. For lab analysis, after proper sampling and refrigeration, samples must be submitted within 4 hours of collection with prior laboratory notification.

(10) 2 mL of 2N Zinc Acetate followed by dropwise addition of 5% Sodium Carbonate solution until precipitation complete.

(11) ISE = Ion Selective Electrode

TABLE 3 - SPECIFIC PARAMETER INFORMATION (Cont'd)

Parameter	Container	Preservation Technique	Minimum Volume Required	Comment
PESTICIDES				
Carbamate Insecticides/Herbicides	Brown glass (1 L)	Refrigerate	Fill container to mark, approx. 900 mL	Scan
Chlorinated Aromatics (12)	A special solvent rinsed container is required and is available through Central Stores.	"	"	Scan
Chlorophenoxy Acid Herbicides (13)		"	"	Scan
Chlorophenols (13)		"	"	Scan
Organochlorine Pesticides		"	"	Scan
Organophosphorus Insecticides		"	"	Scan
Phenyl Urea Herbicides (12)				Scan
Triazine Herbicides		"	"	Scan

(12) Available on a low volume, by special arrangement only.

The Trace Organics Section will, by special arrangement, undertake a low volume of custom miscellaneous pesticide analyses where the above listed classes are inappropriate to the data user's needs. The section is also developing methods to cover a wide range of industrially produced organic contaminants, and invites inquiries from interested samplers.

(13) Special pack #3; PCB bottles with Teflon liners in caps are required.

TABLE 3 - SPECIFIC PARAMETER INFORMATION (Cont'd)

Parameter	Preferred Sampling Container	Preservation Technique	Minimum Volume of Sample (mL)	Analytical Technique
MICROBIOLOGY				
Fecal Pollution Indicators				
Routine				
Total Coliforms	Presterilized glass bottles containing sodium thiosulphate	Refrigeration;	200	Membrane Filtration Incubation on Selective Agar
Fecal Coliforms	"	"	"	"
Fecal Streptococci	"	"	"	"
Presence Absence Procedure	"	"	"	"
Pseudomonas *aeruginosa*	"	"	"	"
Non-routine				
Enterococci	"	"	"	"
**Escherichia* *coli*	"	"	"	"
**Salmonella* sp.	"	"	500 - 1000	Membrane Filtration or Moore Swabs - Incubation In Selective Broth
Organic Enrichment Indicators				
*Fungi	"	"	150	Membrane Filtration Incubation on Selective Agar
Heterotrophic Bacteria				
- Surface Water	"	"	"	Spot or Spread Plate Incubation on Non-Selective Agar
- Treated Water	"	"	"	Membrane Filtration Incubation on Non-Selective Agar
Nuisance Organisms	"	"	"	Membrane Filtration Direct Microscopy or MPN Incubation in Selective Broth
Taxonomy	"	"	"	Direct Microscopy and Biochemical Testing

* Only by special arrangement with the appropriate laboratory.

TABLE 3 - SPECIFIC PARAMETER INFORMATION (Cont'd)

Parameter	Preferred Sampling Container	Preservation Technique	Minimum Volume of Sample (mL)	Analytical Technique
MICROBIOLOGY				
Specific (Industrial/Agricultural) Pollution Indicators				
*Klebsiella sp.	Presterilized glass bottles containing sodium thiosulphate	Refrigeration: for chlorinated samples (see text)	200	Membrane Filtration Incubation on Selective Agar
*Phenol degrading bacteria	"	"	"	MPN Incubation in Selective Broth
*Nitrogen Cycle Bacteria				
- Nitrosomonas sp.	"	"	"	"
- Nitrobacter sp.	"	"	"	"
- Denitrifying Bacteria	"	"	"	"
*Sulphate Reducers	"	Refrigeration only	"	"
*Sulphur Oxidizers	"	"	"	"

Specialized Capabilities

Techniques for collection, handling and analysis of samples will be determined by project needs and must be decided by consultation with the Microbiology Units. Methods are available to determine microbial biomass, metabolic activity transformation reactions, and to identify unknown bacterial isolates.

* Only by prior arrangement with the appropriate laboratory.

TABLE 3 - SPECIFIC PARAMETER INFORMATION (Cont'd)

Type of Test	Container	Preservation Technique	Minimum Volume Required	Comment
AQUATIC BIOLOGY*				
Phytoplankton Biomass (Quantitative)	1 L Glass	Lugol's Iodine Solution - 2 mL/L	1 L	use special plastic lined caps - Pack #3's
Phytoplankton Biomass (Qualitative - Identification)	1 L Glass	Lugol's Iodine Solution and/or Refrigerate - 2 mL/L	1 L	preserved and/or live samples
Algae Identifications algal mass and/or slime accumulations	wide mouth container	Lugol's solution, Formaldehyde and/or refrigerate - 2 mL/L	100 mL	Biological material should only be 1/10 of container volume topped with water
Nuisance Organisms algae, zooplankton, aquatic weeds, pine pollen, shoreline accumulations	wide mouth container	Lugol's solution, Formaldehyde and/or refrigerate - 2 mL/L	100 mL	Biological material should only be 1/10 of container volume topped with water

* Water Resource Branch

TABLE 3 - SPECIFIC PARAMETER INFORMATION (Cont'd)

Type of Test	Sample Size - Grams	
	Minimum	Optimum
TESTS PERFORMED BY THE SEDIMENT & SOILS LABORATORY		
Physical		
Partical Size Distribution:		
Small Size	1	5
Large Size	50	100
Loss on Ignition	2	5
Moisture Content	5	10

Note: For Non-routine tests it is usually advisable to consult with Sediment & Soil Laboratory staff (248-3346).

TABLE 3 - SPECIFIC PARAMETER INFORMATION (Cont'd)

Type of Test	Sample Size - Grams		Extracting Reagent	Parameters	Notes
	Minimum	Optimum			
TESTS PERFORMED BY THE SEDIMENT & SOILS LABORATORY					
Chemical					
Hot Acid Extraction (Routine)	0.2	2 +	HCl/HNO_3	Most metals	Determined by AAS and ICAP
			H_2SO_4/HNO_3	Silver, titanium	
			HCl/HNO_3	Most metals	Ashed vegetation
			H_2SO_4/persulphate	Total nitrogen & phosphorus	
			HCl (after ashing)	Potassium, sodium	
Total Metals (Non-Routine)	1	5 +	$HNO_3/HClO_4/HF/H_2O_2$	Most metals (Not silica)	Also determinations by powde spectrograph, including silica
Chemical Oxygen Demand	0.2	1	$H_2SO_4/K_2Cr_2O_7$		
Extractable Metals	5		DTPA	Some toxic metals (except lead)	Tentative
Mild Extraction Conditions (Non-routine)			Ammonium acetate	Some toxic metals	
			Acetic acid	Some toxic metals	
			Sulphuric acid	Metals, phosphorus	
			Hydrochloric acid	Metals	Sediments
			Water - cold	Anions, Metals & Nutrients Conductivity, pH	
			Water - hot	Boron	
			Copper chloride	Aluminum	Tentative
			Calcium chloride	pH	Preferred method
			Sodium bicarbonate	Phosphorus	Measure of "available" P
Sequential Extraction (Non-routine)	5	20 +	Citrate-dithionite-bicarbonate	Apatite and non-apatite phosphorus	Measure of "available" P
			NaOH		
			HCl		
Leachate (Non-routine)	10	100 +	Water	All	Column and "shake" leach tests
			Dilute acid	All	Usually "shake" tests
			Organic	All	Usually "shake" tests

TABLE 4 - HIGH PRIORITY SUBMISSION PROTOCOL

Samples are designated as "EMERGENCY" when collected as the result of a contingency which poses a direct threat to human health or which may lead to an emergency environmental situation.

Samples must be collected according to recommended laboratory procedures.

Each MOE Region and Branch must designate a senior staff member and alternate person who will co-ordinate high priority submissions for their specific area.

High priority submissions must be authorized by this senior staff member and receipt of high priority samples must be preceded by a phone call to either the central laboratory emergency response co-ordinator or to any appropriate section Emergency Response Task Force (ERTF) representative or Manager (Table VI).

The laboratory personnel contacted will obtain a full description of the problem and sampling details and will ensure that the samples are received when and as described. The contact person is also responsible for ensuring that sample reception staff are notified and that the submission is assigned an "H" priority, and for contacting ERTF representatives in each affected laboratory section. An estimated completion data will be established and transmitted to the client.

All samples must be submitted through LIS with Submission and Request for Analysis forms completed and included with the samples if possible, or forwarded as soon as possible. A signed authorization form from the senior regional co-ordinator must also accompany samples or be forwarded as soon as possible. Forms with photocopied signatures are not acceptable.

Submissions not meeting these requirements will be designated as Normal (N) priority and treated as such.

If arrangements have been made with ERTF personnel, results will be phoned-in to the client immediately upon completion of specific analyses prior to printing of a final LIS report.

THE ERTF will meet every 2 to 3 weeks to review the status of all high priority submissions and to expedite completion of analyses within the estimated time limit.

If the laboratory cannot complete the analysis within the defined time limit, the ERTF representative of the section responsible for the delay must contact the client to explain the problem and assume responsibility for ensuring completion of the work by a mutually agreeable date.

TABLE 4 - HIGH PRIORITY SUBMISSION PROTOCOL EMERGENCY RESPONSE TASK FORCE (ERTF) LABORATORY SERVICES BRANCH

(all area codes are 416)

Central Co-ordinator	Rusty Moody	235-5866

ERTF REPRESENTATIVES

Water Quality	Vera Turner	235-5873
Inorganic Trace Contaminants	Liz Pastorek	235-5855
Trace Organics	Joe Osborne	235-5759
Drinking Water Organics	David Hall	235-5910
	Vince Taguchi	235-5902

SECTION MANAGERS

Water Quality	Larry Vlassoff	235-5805
Inorganic Trace Contaminants	Barry Loescher	235-5848
Trace Organics	George Crawford	235-5757
Drinking Water Organics	Helle Tosine	235-5906
Senior Environmental Scientist (Organics)	Otto Meresz	235-5762

LEGAL SAMPLES

Co-ordinator	Rusty Moody	235-5866
	Barbara O'Neil	235-5965
	Cliff Lee	235-5851

TABLE 5: REGULATION 309 TESTS

TEST	TEST CODE	UNITS
REACTIVE SULPHIDES	RESS	µg/g
REACTIVE CYANIDES	RECN	µg/g
CORROSIVITY	CORVTY	mm/yr
HEAT OF COMBUSTION	HEAT 2	Joules
SLUMP	SLUMP	mm
OIL & GREASE	OANDG	%
CONDUCTIVITY	CONDVTY	µS/cm
MOISTURE	MOIST	%
IGNITABILITY (FLASHPOINT)	IGNIT	°C
pH	PH	
TOTAL RESIDUE, ASHED*	RSTA	%

* Optional

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